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# DUST EXPLOSION SENSITIVITY TESTS ON M-1, M-30, COMPOSITION B, AND HMX

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An experimental program has been conducted to establish the dust explosion sensitivity of M-1, M-30, Composition B and HMX as a function of particle size, dust cloud concentration, and relative humidity. Values of minimum concentration for explosion, minimum ignition energy and minimum ignition temperature have been determined under ambient conditions for three particle sizes. In addition, the effects of relative humidity and concentration on ignition energy have been observed. (cont)

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20. Abstract (Continued)

It has been found that the order of decreasing sensitivity of these materials is as follows: M-1, M-30, Composition B and HMX. The minimum ignition energy was found to increase with increasing particle size, test chamber relative humidity and decreasing dust cloud concentration. Minimum ignition temperature increased with increasing particle size.

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TABLE OF CONTENTS

# SUMMARY

An experimental program has been performed to investigate the effects of particle size, dust cloud concentration and relative humidity on the minimum concentration for explosion, minimum ignition energy and minimum ignition temperature of M-1, M-30, Composition B and HMX propellants and explosives.

Analysis of the results presented in Tables 6, 7 and 8 reveals that the material most sensitive to thermal and electrical initiation sources was M-1. M-30 was the second most sensitive, followed by Composition B and HMX. The minimum ignition energy was observed to increase with increasing particle size, test chamber relative humidity, and decreasing dust cloud concentration. Minimum ignition temperature increased with increasing particle size.

It was also observed that as the particle size of M-1 and M-30 decreased below the 74 micron range, an electrostatic charging phenomenon caused the particles to agglomerate. This resulted in an increase in the minimum concentration for explosion for the smaller particle size ranges.

The data generated on this program should be used by the design engineer in designing future production facilities and by the safety engineer for his evaluation of existing production plants. An outgrowth of this effort could be the development of a monitoring device that senses a dispersed dust cloud, compares its concentration to that required for ignition and takes appropriate action if a hazardous condition exists.

## INTRODUCTION

The objectives of this program were to investigate the effects of particle size, dust cloud concentration and relative humidity of the process air environment on the dust explosion characteristics of HMX, Composition B, M-1 and M-30 explosives and propellants. Specifically, the program was divided into three phases. The objectives of these phases were:

Phase 1

a. Determine the effect of particle size on the minimum concentration for explosion at ambient air conditions.

b. Determine the effect of particle size on the minimum spark ignition energy under ambient air conditions at five times the minimum concentration for explosion.

c. Determine the effect of particle size on the minimum ignition temperatures of HMX, Composition B, M-1 and M-30 dust clouds.

Phase 2

Determine the effect of temperature, particle size and relative humidity on the minimum ignition energy of the energetic materials at five times their minimum concentration for explosion.

Phase 3

Determine the effect that reduced dust loadings have on the minimum spark ignition energies obtained in Phase 2. Evaluate these energies for various particle sizes and dust concentrations of three and one times the minimum concentration for explosion of the respective materials.

The introduction of the concept of "continuous" vs. "batch" production of sensitive materials has resulted in a marked increase in the requirement for dust control equipment. Higher production rates require the use of conveyor belts, fluidized bed dryers, continuous casting belts, automatic weighing and bagging machines, vibratory and weigh feeders. Proper design of dust control equipment requires a knowledge of the particle size distribution of the material being processed and the effects environmental conditions have upon the dust explosion characteristics of the materials being produced.

Hazards Analysis requirements of ARMCOM Regulation No. 385-4 specify that all hazards be classified. This classification is based on a comparison of the mechanical, electrical and thermal input

energies that exist, due to normal process operations, with the sensitivity to initiation of the explosive material (threshold initiation level). Extensive investigation into the literature failed to uncover adequate data on the ignition sensitivities of various forms of the materials selected for evaluation. In addition, the existing data banks did not provide detailed information on the effects of particle size, concentration, air temperature and relative humidity on the ignition sensitivities of these materials.

## DESCRIPTION OF EXPERIMENTS

### Phase 1

The experiments performed during this phase of the program utilized three standard types of laboratory equipment developed by the Bureau of Mines, United States Department of Labor, namely:

1. Hartmann apparatus for determining the minimum spark ignition energy for ignition of a dust cloud.

2. Hartmann apparatus for determining the minimum concentration for explosion of a dust cloud.

3. Godbert-Greenwald furnace for determining the minimum ignition temperature of a dust cloud.

#### Minimum Spark Ignition Energy of a Dust Cloud

The minimum electrical energy required to ignite a dust cloud is determined in the Hartmann apparatus. This apparatus is shown schematically in Figure 1 and pictorially in Figure 2. It consists of a vertically mounted, seven cm diameter combustion tube, 30.5 cm long and auxiliary equipment for producing the dust dispersion. The tube, made of Lucite, is attached to a cylindrical metal base (dispersion cup) by four brass bolts. Figure 3 is a photograph which shows a close-up of the Lucite tube and dispersion cup which are both wrapped with nichrome heating wire. The top of the tube is covered with a filter paper diaphragm held in place by a locking ring. Figure 4 is a close-up view of the brass dust dispersion cup containing the mushroom shaped air deflector. Nichrome heating wires are shown wrapped around the base. It is seen that the top surface of the base is hemispherically shaped. The total free volume of the test chamber is 1.23 liters. Dispersion is accomplished by a single blast of air from the 1.31 liter reservoir shown in Figure 5. Air pressure in the reservoir is 69 kilopascals (kPa). The quantity of dust dispersed is five times the minimum explosive concentration or a maximum of 2 gm/liter. Concentrations greater than 2 gm/liter cannot be dispersed in this apparatus.

The igniting spark passes between two pointed, 20 gauge tungsten electrodes that are separated by a 0.64 cm air gap. These electrodes are mounted 10 cm above the base of the tube. Electrical energy for the spark ignition is obtained from the discharge of condensers at 100 or 400 volts. The bank of ten condensers has a capacitance range of 2 to 100 microfarads. This combination of voltage and capacitance allows energy levels to be varied from 50 to 500 millijoules in 50 millijoule increments (at 100 volts) and from 800 to 8000 millijoules in 800 millijoule increments (at 400 volts).

The control console that houses the electronics necessary to provide the variable electrical discharge energy is shown in Figure 6.

The energy of the spark (in joules) is calculated as $0.5\,CV^2$, where C is the capacitance of the condensers in farads, and V is the charging potential in volts. Dust cloud minimum ignition energy is the least amount of energy required to produce flame propagation of 10 cm or longer in the tube. Four trials are made at each condenser setting; however, if the dust ignites in initial trials, lower energy is tried until a minimum is obtained. A typical dust explosion in the Lucite tube test chamber is shown in Figure 7.

### Minimum Explosive Concentration

The minimum explosive concentration or the lower explosive limit of a dust sample is determined in the previously described Hartmann apparatus, except that an induction spark igniting source is used instead of the timed condenser discharge spark. A weighed amount of dust is spread in a thin layer in the dispersion cup. The top of the Hartmann tube (Lucite) is covered with a filter paper diaphragm held in place by a locking ring. A 0.16 cm hole is made in the center of the filter paper to prevent pressure build-up in the tube from the dispersing air and the tungsten electrodes are adjusted to a gap length of 0.48 cm. The electric spark is struck and the current adjusted to 23.5 milliamperes. The dust cloud is formed in the Lucite tube by releasing air from the 1.31 liter reservoir through the full-port solenoid valve; optimum air pressure is 69 kPa.

Following ignition of the dust, sufficient pressure must develop to burst the filter paper diaphragm; appearance of flame in the tube is not considered propagation. The pressure required to burst the paper diaphragm is about 21 kPa, depending on the rate of pressure rise. If propagation occurs for a given weight of dust, the weight is reduced by a five-milligram increment and another trial made until a quantity is obtained which fails to propagate flame in any of four successive trials. The lowest weight at which flame propagates is used in calculating the minimum concentration. Tests are made with the electrodes 10 cm from the bottom of the tube. Figure 8 is a schematic of the Hartmann apparatus for determining the minimum concentration for explosion of a dust cloud.

### Minimum Ignition Temperature of a Dust Cloud

The minimum ignition temperature of a dust cloud is determined in the Godbert-Greenwald Furnace. This furnace consists of a 3.7 cm diameter vertical alundum tube, 23 cm long, wound with 6.4 meters of 18-gauge nichrome V wire. The windings are placed closer together toward the two ends than in the middle of the tube to obtain relatively even temperature throughout. The tube is mounted between two 1.3 cm thick transite plates in a 15 cm diameter sheet

metal cylinder with kieselguhr packing between the alundum tube and the sheet-metal shell. A glass adapter connects the top of the tube to a small brass chamber that has a hinged lid for inserting the dust. A full port solenoid valve between the dust chamber and a 500-milliliter air reservoir controls the dispersion of the dust. The air reservoir is connected to a compressed air line and a pressure gauge. Dust contained in the brass chamber is dispersed downward through the furnace by a single blast of the compressed air from the reservoir. Dispersion pressure is 21 kPa. The weight of sample placed in the brass chamber is varied in accordance with the concentration desired in the furnace. This apparatus is depicted schematically and pictorially in Figures 9 and 10.

The temperature in the furnace is measured with a 22-gauge chromel-alumel thermocouple which is located 0.08 cm from the furnace wall at mid-height. An automatic temperature controller maintains the furnace temperature at the desired value. Ignition is denoted by the appearance of flame below the mouth of the furnace. The ignition temperature is the minimum furnace temperature at which flame appears at the bottom of the furnace in one or more trials in a group of four. Furnace temperature is varied in 10°C increments; the highest temperature attained in the furnace is 750°C.

Phase 2

This phase of the program required that the Hartmann apparatus be modified to allow for the control of air temperature and relative humidity. A complete description of this modification is presented in Appendix B under the test air environmental control methods section. Dust cloud concentrations were maintained at five times the minimum concentrations for explosion established in Phase 1. Once again the criterion for selecting the threshold minimum ignition energy level was four negative results below the last positive result for either a 0.05 or 0.80 joule energy interval.

The environmental test air conditions for the Hartmann apparatus on this phase of the program were 24°C at 2, 50 and 76% relative humidities and 52°C at 2, 39 and 58% relative humidities.

Phase 3

The unmodified Hartmann apparatus was used on this phase of the program to determine the effect of dust concentration on minimum ignition energy. Dust concentrations of three and one times the minimum concentration for explosion were used. Test air was maintained at 24°C and one percent relative humidity. Previously described test procedures were followed.

## EXPERIMENTAL RESULTS

Tables 1 through 3 present the results of the tests performed on phase 1 of this program while Tables 4 and 5 present results for phases 2 and 3 respectively. Figures 11 through 32 graphically present the data contained in all five tables.

### Phase 1

#### Minimum Explosive Concentration

Results of this test series are presented in Table 1 and Figures 11 and 12. It is seen that 200 mesh M-1 has the lowest minimum concentration for explosion followed by 200 mesh M-30, 100 mesh Comp B and classes 1 and 3 HMX. Figure 11 shows that both M-1 and M-30 had their highest minimum concentrations for explosion at the smallest particle sizes. This phenomenon is explained by the observed tendency for the 44 micron particles to hold an electrostatic charge and agglomerate. The net effect is that they cling to themselves and behave as much larger particles in the test apparatus.

Composition B appears to behave differently from the two propellants. Its minimum concentration for explosion decreases as its particle size increases. This is also attributed to the electrostatic charging phenomenon. HMX reaches its lowest value of minimum concentration for explosion at classes 1 and 3 particle sizes.

#### Minimum Ignition Energy of a Dust Cloud

Table 2 presents the results of this series of tests. It is apparent that M-1 and Comp B are the two most sensitive materials followed by M-30 and HMX. Figures 13 and 14 show M-1, M-30 and Composition B all plateau out at the 44 and 74 micron particle size ranges. As expected, the highest minimum ignition energies occur at the largest particle sizes for all four materials. The general rule that the minimum ignition energy increases with increasing sample particle size is followed in this case.

#### Minimum Ignition Temperature of a Dust Cloud

Results of the minimum ignition temperature tests for dispersed dust clouds of the four sample materials are presented in Table 3. Variations in ignition temperature as a function of particle size and dust cloud concentration are presented. Figures 15 and 16 graphically illustrate the effect of particle size on ignition temperature for a fixed dust cloud concentration. M-1 has the lowest minimum ignition temperature (210°C) followed by M-30 (230°C). Both of these minimums occurred at the 44 micron particle size range. Composition B ignites at 360°C and 44 micron particle size. Reducing the loading by 0.1 gm/l resulted in a 30°C drop in ignition temperature.

Class 1 HMX ignited at 350°C at a 0.92 gm/l concentration. It is apparent from the broad ranges of ignition temperatures found in Figure 16 that the ignition temperature of HMX is very sensitive to particle size and dust concentration. The spread of ignition temperatures for HMX lies between 350°C and 750°C.

Phase 2

Minimum Ignition Energy, Controlled Environmental Air

Table 4 presents the results of the experimental effort performed on this phase of the program. Figures 17 through 24 present minimum ignition energy as a function of relative humidity for the four samples evaluated. Figures 25 through 28 show the effect of particle size on minimum ignition energy. Careful study of Figures 17 and 18 reveals that the trend is for the minimum ignition energy of M-1 propellant to increase with increasing relative humidity. The lowest value of minimum ignition energy for M-1 always occurs at the 2 percent relative humidity point. Variations inignition energy are significant between humidity levels at fixed temperatures.

Figures 19 and 20 present the results for M-30 propellant. The same trend is seen to exist, increased ignition energy at increased relative humidity. A minimum value of ignition energy occurs at the 44 micron particle size range at the 2 percent relative humidity level. Comparison of M-1 and M-30 data reveals that the M-1 is more sensitive (0.20 joules) than the M-30 (0.30 joules) at the 2 percent relative humidity values.

Composition B results plotted in Figures 21 and 22 follow the same pattern established by the two propellants. The lowest value of ignition energy (0.20 joules) occurred at the finest particle size, 44 microns, and the 2 percent relative humidity value. Relative humidity and temperature affect the results by factors of up to 20 times the minimum value of 0.20 joules.

The results of the minimum ignition energy tests on HMX are presented in Figures 23 and 24. Values of ignition energy greater than 8 joules were plotted as 8 joules for lack of a better way of presenting the data. A general trend exists for the ignition energy to increase with increasing relative humidity. The lowest value of 1.60 joules occurred at the 2 percent relative humidity value for the class 1 material. Throughout this report it is noted that the HMX data is the least consistent of the four materials tested. This is attributed to the broad range of particle sizes found in the classes 1 and 3 material (see Table B-1). The reproducibility of each data point is dependent upon the particle size distribution of each test sample. The broader that this distribution is, the more random will be the results of the ignition energy tests.

Figure 25 presents plots of minimum ignition energy versus particle size for M-1 propellant at two temperatures and three relative humidities. It is seen that for the 52°C data the lowest ignition energies, for a given relative humidity curve, occur at the 74 micron particle size range. Differences between the 39 and the 2 percent relative humidity values disappear at the 149 micron particle size range. Examination of the 24°C graph reveals that the highest ignition energy values occurred at the 74 micron particle size point. This result is contrary to all of the other data observed in this phase of the program. The reason for this discrepancy is not known.

Results of the tests on M-30 are presented in Figure 26. The trend is for the minimum ignition energy to increase with increasing particle size. Definite differences in results due to relative humidity are readily apparent. In general, increasing the relative humidity and particle size increases the minimum ignition energy.

Composition B test results are shown in Figure 27. Particle size has little effect in the 44 to 74 micron range. Above these values, the ignition energy increases substantially. Looking at the 24°C graph, it is seen that the minimum energy increases from 0.20 joules at 2 percent relative humidity to 1.60 joules at 76 percent relative humidity. This is significant because in a process operation it is likely that an ungrounded, conductive element can store and discharge 0.20 joules. However, it is highly unlikely that the same piece of equipment will be able to accumulate 1.60 joules of electrical energy.

Figure 28 shows the variation in minimum ignition energy with particle size for HMX at two air temperatures and three relative humidities. The lowest values of ignition energy occur at the class 1 points. It is seen that the most sensitive condition (1.6 joules) occurs at the class 1 size at 2 percent relative humidity and 24°C air temperature.

## Phase 3

### Minimum Ignition Energy at Reduced Concentrations

Table 5 presents the results of the test effort expended on this phase of the program. Figures 29 through 32 graphically illustrate the data contained in the table. All of the data in these four figures have the same characteristic. Namely, the minimum ignition energy increases markedly as the concentration is decreased from 5 to 3 to 1 times the minimum concentration for explosion. For example, M-1 at 149 micron particle size has a minimum ignition energy of 0.25 joules at 0.25 gm/l. This value rises to 1.6 joules at 0.15 gm/l and is greater than 8 joules at 0.05 gm/l (the minimum concentration for explosion). The significance of this finding is readily apparent in hazards analysis work. It is currently common practice to evaluate dust explosion hazards by calculating the electrical energy available

in a given process operation and comparing this value with the minimum ignition energy of the material. No allowance is made for the fact that the minimum ignition energy is based on concentrations that are 5 to 10 times the minimum concentration for explosion. This oversight introduces factors of safety which are so large that they make the calculation of the probability of the event occurring a fruitless exercise. Data presented in this report should be useful to the hazards analysis investigator because it will allow him to more realistically evaluate a specific situation.

## CONCLUSIONS

As a result of the dust phase explosion tests performed on this program on M-1, M-30, Composition B and HMX it is possible to conclude the following:

1. M-1 has the lowest minimum concentration for explosion followed by M-30, Comp B and HMX.

2. Particles of M-1 and M-30 below the 74 micron range tend to agglomerate and cling to themselves due to an electrostatic charging phenomenon. The result is that they yield higher minimum concentrations for explosion than the 74 micron particles.

3. The minimum ignition energy increases with increasing sample particle size. M-1 and Comp B are the two most sensitive to electrostatic discharge followed by M-30 and HMX.

4. As the particle size increases, the minimum ignition temperature increases. M-1 has the lowest minimum ignition temperature followed by M-30, Comp B and HMX.

5. The minimum ignition energy of all four materials increases with increasing test chamber relative humidity. Lowest values of ignition energy occurred at the 2 percent relative humidity point for each of the four materials tested.

6. The minimum ignition energy of the four materials increases markedly as the dust cloud concentration is decreased from 5 to 3 to 1 times the minimum concentration for explosion.

7. Hazards analysis of electrostatic discharge potentials in a given process must be based upon consideration of the effect of concentration on minimum ignition energy (current practice is to use ignition energies which are obtained at 5 to 10 times the minimum concentration for explosion).

## RECOMMENDATIONS

It is recommended that implementation of the following items be considered:

1. Results obtained from this program should be incorporated into a standard hazards analysis data bank.

2. Additional explosive and propellant materials should be tested for the effects of particle size, dust cloud concentration and relative humidity on the minimum concentration for explosion, minimum ignition energy and minimum ignition temperature.

3. Obtain dust samples from in-process operations at Army Ammunition Plants and establish the potential explosion hazard by comparing the sampled concentrations with the measured data generated by this program.

Table 1. Results of minimum concentration for explosion tests

| Sample | U.S. sieve number[a] | Minimum explosive concentration (gm/l) |
|---|---|---|
| M-1 | 100 | 0.05 |
| | 200 | 0.04 |
| | 325 | 0.06 |
| M-30 | 100 | 0.07 |
| | 200 | 0.05 |
| | 325 | 0.12 |
| Comp B | 100 | 0.06 |
| | 200 | 0.08 |
| | 325 | 0.09 |
| HMX | Class 1[b] | 0.50 |
| | Class 3 | 0.50 |
| | Class 5 | 1.00 |

[a]Material passed through the sieve indicated.
[b]Appendix A defines class particle size designations.

Table 2. Results of minimum ignition energy of du. t cloud tests

| Sample | U. S. sieve number[a] | Concentration (gm/l) | Min. ign. energy Go (joules) | Min. ign. energy No Go (joules) |
|---|---|---|---|---|
| M-1 | 100 | 0.25 | 0.25 | 0.20 |
| | 200 | 0.20 | 0.20 | 0.15 |
| | | 0.15 | 0.20 | 0.15 |
| | 325 | 0.30 | 0.20 | 0.15 |
| | | 0.25 | 0.20 | 0.15 |
| M-30 | 100 | 0.50 | 0.50 | 0.45 |
| | | 0.35 | 0.40 | 0.35 |
| | 200 | 0.25 | 0.25 | 0.20 |
| | 325 | 0.60 | 0.25 | 0.20 |
| Comp B | 100 | 0.50 | 0.45 | 0.40 |
| | | 0.30 | 1.60 | 0.80 |
| | | 0.25 | 1.60 | 0.80 |
| | 200 | 0.40 | 0.20 | 0.15 |
| | | 0.35 | 0.20 | 0.15 |
| | 325 | 0.45 | 0.20 | 0.15 |
| | | 0.40 | 0.20 | 0.15 |
| HMX | Class 1[b] | 2.00 | 0.45 | 0.40 |
| | Class 3 | 2.00 | 5.60 | 4.80 |
| | Class 5 | 2.00 | 4.00 | 3.20 |

[a]Material passed through the sieve indicated.
[b]Appendix A defines class particle size designations.

Table 3. Results of minimum ignition temperature of dust cloud tests

| Sample | U. S. sieve number[a] | Concentration (gm/l) | Minimum ignition temperature (°C) |
|---|---|---|---|
| M-1 | 100 | 0.46 | 220 |
| | | 0.37 | 230 |
| | 200 | 0.37 | 240 |
| | | 0.27 | 240 |
| | 325 | 0.55 | 210 |
| | | 0.46 | 210 |
| M-30 | 100 | 0.92 | 250 |
| | | 0.64 | 250 |
| | 200 | 0.92 | 230 |
| | | 0.64 | 240 |
| | 325 | 0.92 | 230 |
| | | 0.64 | 230 |
| Comp B | 100 | 0.92 | 390 |
| | | 0.55 | 330 |
| | | 0.46 | 430 |
| | 200 | 0.92 | 360 |
| | | 0.73 | 350 |
| | | 0.64 | 380 |
| | | 0.46 | 430 |
| | 325 | 0.92 | 360 |
| | | 0.83 | 330 |
| | | 0.73 | 360 |
| HMX | Class 1[b] | 1.38 | 380 |
| | | 0.92 | 350 |
| | Class 3 | 1.38 | 690 |
| | | 0.92 | >750[c] |
| | Class 5 | 1.38 | 680 |
| | | 0.92 | >750 |

[a] Material passed through the sieve indicated.
[b] Appendix A defines class particle size designations.
[c] The Godbert-Greenwald furnace could not be heated past 750°C.

Table 4. Results of minimum ignition energy tests under controlled environmental air conditions

| Sample | U. S. sieve number[a] | Concentration (gm/l) | Temperature (°C) | Relative humidity (%) | Minimum ignition energy Go (joules) | Minimum ignition energy No Go (joules) |
|---|---|---|---|---|---|---|
| M-1 | 100 | 0.25 | 52 | 58 | 1.60 | 0.80 |
| | | 0.25 | 52 | 39 | 0.40 | 0.30 |
| | | 0.25 | 52 | 2 | 0.40 | 0.30 |
| | | 0.25 | 24 | 76 | 0.40 | 0.30 |
| | | 0.25 | 24 | 50 | 0.40 | 0.30 |
| | | 0.25 | 24 | 2 | 0.20 | 0.10 |
| | 200 | 0.20 | 52 | 58 | 0.40 | 0.30 |
| | | 0.20 | 52 | 39 | 0.30 | 0.20 |
| | | 0.20 | 52 | 2 | 0.20 | 0.10 |
| | | 0.20 | 24 | 76 | 0.80 | 0.50 |
| | | 0.20 | 24 | 50 | 0.80 | 0.50 |
| | | 0.20 | 24 | 2 | 0.30 | 0.20 |
| | 325 | 0.30 | 52 | 58 | 0.50 | 0.40 |
| | | 0.30 | 52 | 39 | 0.40 | 0.30 |
| | | 0.30 | 52 | 2 | 0.20 | 0.10 |
| | | 0.30 | 24 | 76 | 0.50 | 0.40 |
| | | 0.30 | 24 | 50 | 0.40 | 0.30 |
| | | 0.30 | 24 | 2 | 0.20 | 0.10 |

[a] Material passed through the sieve indicated.
[b] Appendix A defines class particle size designations.
[c] The test apparatus could not discharge more than 8.00 joules.

Table 4. Results of minimum ignition energy tests under controlled environmental air conditions (cont.)

| Sample | U.S. sieve number[a] | Concentration (gm/l) | Temperature (°C) | Relative humidity (%) | Minimum ignition energy Go (joules) | No Go (joules) |
|---|---|---|---|---|---|---|
| M-30 | 100 | 0.35 | 52 | 58 | 3.20 | 2.40 |
| | | 0.35 | 52 | 39 | 2.40 | 1.60 |
| | | 0.35 | 52 | 2 | 1.60 | 0.80 |
| | | 0.35 | 24 | 76 | 2.40 | 1.60 |
| | | 0.35 | 24 | 50 | 1.60 | 0.80 |
| | | 0.35 | 24 | 2 | 0.50 | 0.40 |
| | 200 | 0.25 | 52 | 58 | 1.60 | 0.80 |
| | | 0.25 | 52 | 39 | 1.60 | 0.80 |
| | | 0.25 | 52 | 2 | 0.40 | 0.30 |
| | | 0.25 | 24 | 76 | 1.60 | 0.80 |
| | | 0.25 | 24 | 50 | 1.60 | 0.80 |
| | | 0.25 | 24 | 2 | 0.40 | 0.30 |
| | 325 | 0.60 | 52 | 58 | 1.60 | 0.80 |
| | | 0.60 | 52 | 39 | 0.30 | 0.20 |
| | | 0.60 | 52 | 2 | 0.30 | 0.20 |
| | | 0.60 | 24 | 76 | 0.50 | 0.40 |
| | | 0.60 | 24 | 50 | 0.50 | 0.40 |
| | | 0.60 | 24 | 2 | 0.30 | 0.20 |

[a]Material passed through the sieve indicated.
[b]Appendix A defines class particle size designations.
[c]The test apparatus could not discharge more than 8.00 joules.

Table 4. Results of minimum ignition energy tests under controlled environmental air conditions (cont.)

| Sample | U.S. sieve number[a] | Concentration (gm/l) | Temperature ($^{o}C$) | Relative humidity (%) | Minimum ignition energy Go (joules) | No Go (joules) |
|---|---|---|---|---|---|---|
| Comp B | 100 | 0.30 | 52 | 58 | 4.80 | 4.00 |
| | | 0.30 | 52 | 39 | 4.00 | 3.20 |
| | | 0.30 | 52 | 2 | 0.50 | 0.40 |
| | | 0.30 | 24 | 76 | 4.80 | 4.00 |
| | | 0.30 | 24 | 50 | 4.00 | 3.20 |
| | | 0.30 | 24 | 2 | 2.40 | 1.60 |
| | 200 | 0.40 | 52 | 58 | 2.40 | 1.60 |
| | | 0.40 | 52 | 39 | 1.60 | 0.80 |
| | | 0.40 | 52 | 2 | 0.30 | 0.20 |
| | | 0.40 | 24 | 76 | 1.60 | 0.80 |
| | | 0.40 | 24 | 50 | 1.60 | 0.80 |
| | | 0.40 | 24 | 2 | 0.30 | 0.20 |
| | 325 | 0.45 | 52 | 58 | 2.40 | 1.60 |
| | | 0.45 | 52 | 39 | 1.60 | 0.80 |
| | | 0.45 | 52 | 2 | 0.30 | 0.20 |
| | | 0.45 | 24 | 76 | 1.60 | 0.80 |
| | | 0.45 | 24 | 50 | 0.80 | 0.50 |
| | | 0.45 | 24 | 2 | 0.20 | 0.10 |

[a] Material passed through the sieve indicated.
[b] Appendix A defines class particle size designations.
[c] The test apparatus could not discharge more than 8.00 joules.

Table 4. Results of minimum ignition energy tests under controlled environmental air conditions (cont.)

| Sample | U.S. sieve number[a] | Concentration (gm/l) | Temperature (°C) | Relative humidity (%) | Minimum ignition energy Go (joules) | No Go (joules) |
|---|---|---|---|---|---|---|
| HMX | Class 1[b] | 2.00 | 52 | 58 | 7.20 | 6.40 |
| | | 2.00 | 52 | 39 | 4.80 | 4.00 |
| | | 2.00 | 52 | 2 | 2.40 | 1.60 |
| | | 2.00 | 24 | 76 | 6.40 | 5.60 |
| | | 2.00 | 24 | 50 | 4.80 | 4.00 |
| | | 2.00 | 24 | 25 | 3.20 | 2.40 |
| | | 2.00 | 24 | 2 | 1.60 | 0.80 |
| | Class 3 | 2.00 | 52 | 58 | >8.00[c] | 8.00 |
| | | 2.00 | 52 | 39 | >8.00 | 8.00 |
| | | 2.00 | 52 | 2 | 3.20 | 2.40 |
| | | 2.00 | 24 | 76 | >8.00 | 8.00 |
| | | 2.00 | 24 | 50 | 6.40 | 5.60 |
| | | 2.00 | 24 | 2 | >8.00 | 8.00 |
| | Class 5 | 2.00 | 52 | 58 | 8.00 | 7.20 |
| | | 2.00 | 52 | 39 | 7.20 | 6.40 |
| | | 2.00 | 52 | 2 | >8.00 | 8.00 |
| | | 2.00 | 24 | 76 | >8.00 | 8.00 |
| | | 2.00 | 24 | 50 | >8.00 | 8.00 |
| | | 2.00 | 24 | 2 | 8.00 | 7.20 |

[a] Material passed through the sieve indicated.
[b] Appendix A defines class particle size designations.
[c] The test apparatus could not discharge more than 8.00 joules.

Table 5. Results of minimum ignition energy tests at reduced dust concentrations

| Sample | U. S. sieve number[a] | Concentration (gm/l) | Min. ign. energy* Go (joules) | No Go (joules) |
|---|---|---|---|---|
| M-1 | 100 | 0.15 | 1.60 | 0.80 |
| | | 0.05 | >8.00 | 8.00 |
| | 200 | 0.12 | 0.30 | 0.20 |
| | | 0.04 | 7.20 | 6.40 |
| | 325 | 0.18 | 0.20 | 0.10 |
| | | 0.06 | 2.40 | 1.60 |
| M-30 | 100 | 0.21 | 3.20 | 2.40 |
| | | 0.07 | >8.00 | 8.00 |
| | 200 | 0.15 | 3.20 | 2.40 |
| | | 0.05 | >8.00 | 8.00 |
| | 325 | 0.36 | 0.80 | 0.50 |
| | | 0.12 | 1.60 | 0.80 |
| Comp B | 100 | 0.18 | 2.40 | 1.60 |
| | | 0.06 | >8.00 | 8.00 |
| | 200 | 0.24 | 0.20 | 0.10 |
| | | 0.08 | 2.40 | 1.60 |
| | 325 | 0.27 | 0.20 | 0.10 |
| | | 0.09 | 0.80 | 0.50 |
| HMX | Class 1 | 1.50 | 2.40 | 1.60 |
| | | 0.50 | 6.40 | 5.60 |
| | Class 3 | 1.50 | >8.00 | 8.00 |
| | | 0.50 | >8.00 | 8.00 |
| | Class 5 | 1.50 | >8.00 | 8.00 |
| | | 0.50 | >8.00 | 8.00 |

*All data were obtained using 24°C test air containing one percent relative humidity.

Table 6. Summary of ambient minimum concentration, energy and temperature test results

| Sample | Sieve no. | Min. expl. conc. (gm/l) | Min. ign. en. Conc. (gm/l) | Min. ign. en. Energy (joules) | Min. ign. temp. Conc. (gm/l) | Min. ign. temp. Temp. (°C) |
|---|---|---|---|---|---|---|
| M-1 | 100 | 0.05 | 0.25 | 0.25 | 0.46 | 220 |
| | | | | | 0.37 | 230 |
| | 200 | 0.04 | 0.20 | 0.20 | 0.37 | 240 |
| | | | 0.15 | 0.20 | 0.27 | 240 |
| | 325 | 0.06 | 0.30 | 0.20 | 0.55 | 210 |
| | | | 0.25 | 0.20 | 0.46 | 210 |
| M-30 | 100 | 0.07 | 0.50 | 0.50 | 0.92 | 250 |
| | | | 0.35 | 0.40 | 0.64 | 250 |
| | 200 | 0.05 | 0.25 | 0.25 | 0.92 | 230 |
| | | | | | 0.64 | 240 |
| | 325 | 0.12 | 0.60 | 0.25 | 0.92 | 230 |
| | | | | | 0.64 | 230 |
| Comp B | 100 | 0.06 | 0.50 | 0.45 | 0.92 | 390 |
| | | | 0.30 | 1.60 | 0.55 | 330 |
| | | | 0.25 | 1.60 | 0.46 | 430 |
| | 200 | 0.08 | 0.40 | 0.20 | 0.92 | 360 |
| | | | 0.35 | 0.20 | 0.73 | 350 |
| | | | | | 0.64 | 380 |
| | | | | | 0.46 | 430 |
| | 325 | 0.09 | 0.45 | 0.20 | 0.92 | 360 |
| | | | 0.40 | 0.20 | 0.83 | 330 |
| | | | | | 0.73 | 360 |
| HMX | Class 1 | 0.50 | 2.00 | 0.45 | 1.38 | 380 |
| | | | | | 0.92 | 350 |
| | Class 3 | 0.50 | 2.00 | 5.60 | 1.38 | 690 |
| | | | | | 0.92 | > 750 |
| | Class 5 | 1.00 | 2.00 | 4.00 | 1.38 | 680 |
| | | | | | 0.92 | > 750 |

Table 7. Summary of ignition energy tests, controlled environment

| Sieve no. | Temp. (°C) | R.H. (%) | Minimum ignition energy[a] (joules) M-1 | M-30 | Comp B | HMX[b] |
|---|---|---|---|---|---|---|
| 100 | 52 | 58 | 1.60 | 3.20 | 4.80 | 7.20 |
| | 52 | 39 | 0.40 | 2.40 | 4.00 | 4.80 |
| | 52 | 2 | 0.40 | 1.60 | 0.50 | 2.40 |
| | 24 | 76 | 0.40 | 2.40 | 4.80 | 6.40 |
| | 24 | 50 | 0.40 | 1.60 | 4.00 | 4.80 |
| | 24 | 2 | 0.20 | 0.50 | 2.40 | 1.60 |
| 200 | 52 | 58 | 0.40 | 1.60 | 2.40 | > 8.00 |
| | 52 | 39 | 0.30 | 1.60 | 1.60 | > 8.00 |
| | 52 | 2 | 0.20 | 0.40 | 0.30 | 3.20 |
| | 24 | 76 | 0.80 | 1.60 | 1.60 | > 8.00 |
| | 24 | 50 | 0.80 | 1.60 | 1.60 | 6.40 |
| | 24 | 2 | 0.30 | 0.40 | 0.30 | > 8.00 |
| 325 | 52 | 58 | 0.50 | 1.60 | 2.40 | 8.00 |
| | 52 | 39 | 0.40 | 0.30 | 1.60 | 7.20 |
| | 52 | 2 | 0.20 | 0.30 | 0.30 | > 8.00 |
| | 24 | 76 | 0.50 | 0.50 | 1.60 | > 8.00 |
| | 24 | 50 | 0.40 | 0.50 | 0.80 | > 8.00 |
| | 24 | 2 | 0.20 | 0.30 | 0.20 | 8.00 |

Table 8. Summary of ignition energy tests, reduced dust concentrations

| | Minimum ignition energy | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | M-1 | | M-30 | | Comp B | | HMX[b] | |
| Sieve no. | Conc. (gm/l) | Energy (joules) | Conc. (gm/l) | Energy (joules) | Conc. (gm/l) | Energy (joules) | Conc. (gm/l) | Energy (joules) |
| 100 | 0.15 | 1.60 | 0.21 | 3.20 | 0.18 | 2.40 | 1.50 | 2.40 |
| | 0.05 | > 8.00 | 0.07 | > 8.00 | 0.06 | > 8.00 | 0.50 | 6.40 |
| 200 | 0.12 | 0.30 | 0.15 | 3.20 | 0.24 | 0.20 | 1.50 | > 8.00 |
| | 0.04 | 7.20 | 0.05 | > 8.00 | 0.08 | 2.40 | 0.50 | > 8.00 |
| 325 | 0.18 | 0.20 | 0.36 | 0.80 | 0.27 | 0.20 | 1.50 | > 8.00 |
| | 0.06 | 2.40 | 0.12 | 1.60 | 0.09 | 0.80 | 0.50 | > 8.00 |

[a]See Table 4 for dust concentrations.

[b]Sieve numbers 100, 200 and 325 are replaced by classes 1, 3 and 5 for HMX.

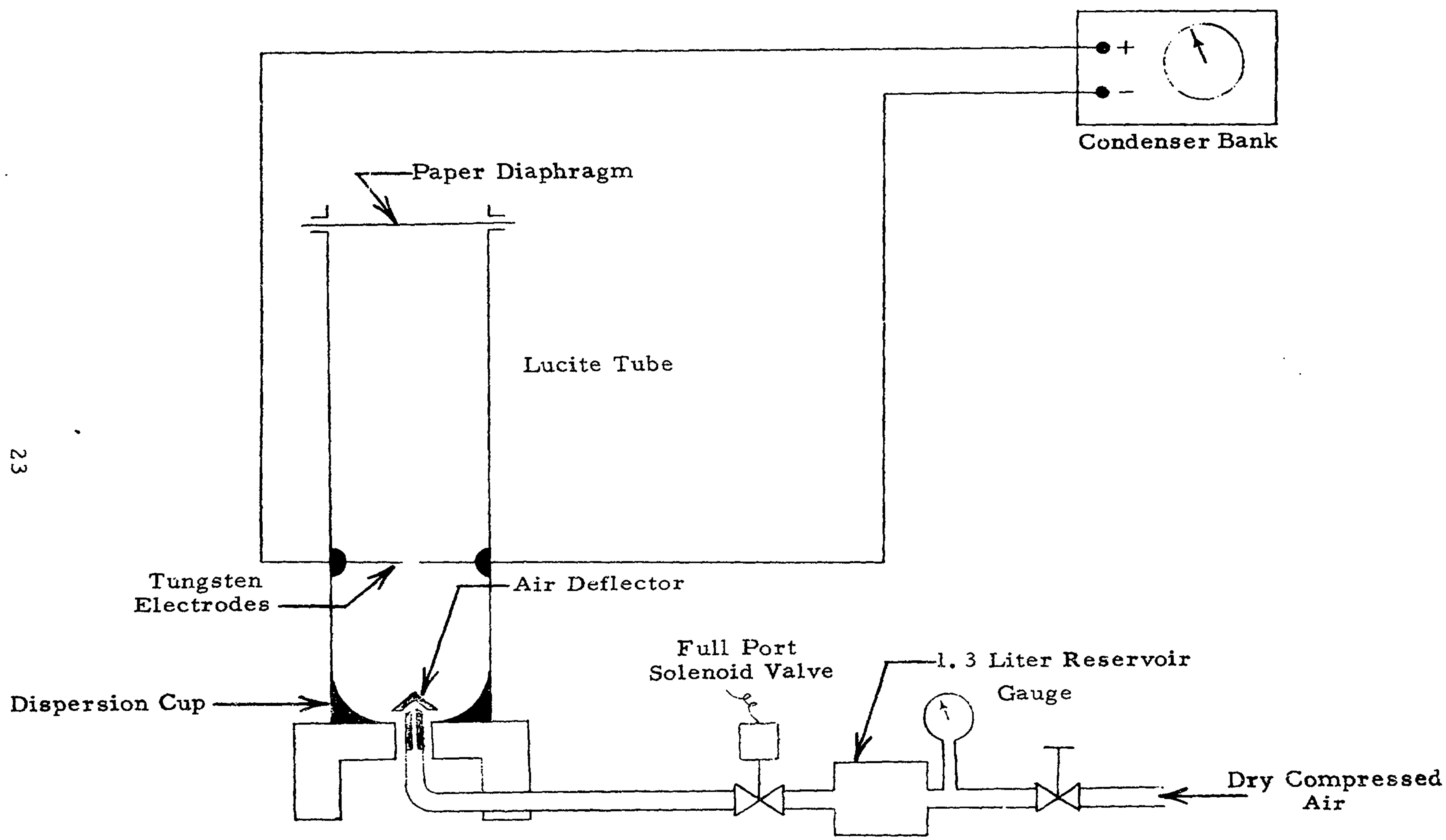


Figure 1. Hartmann apparatus for determining the minimum electrical energy for ignition of a dust cloud.

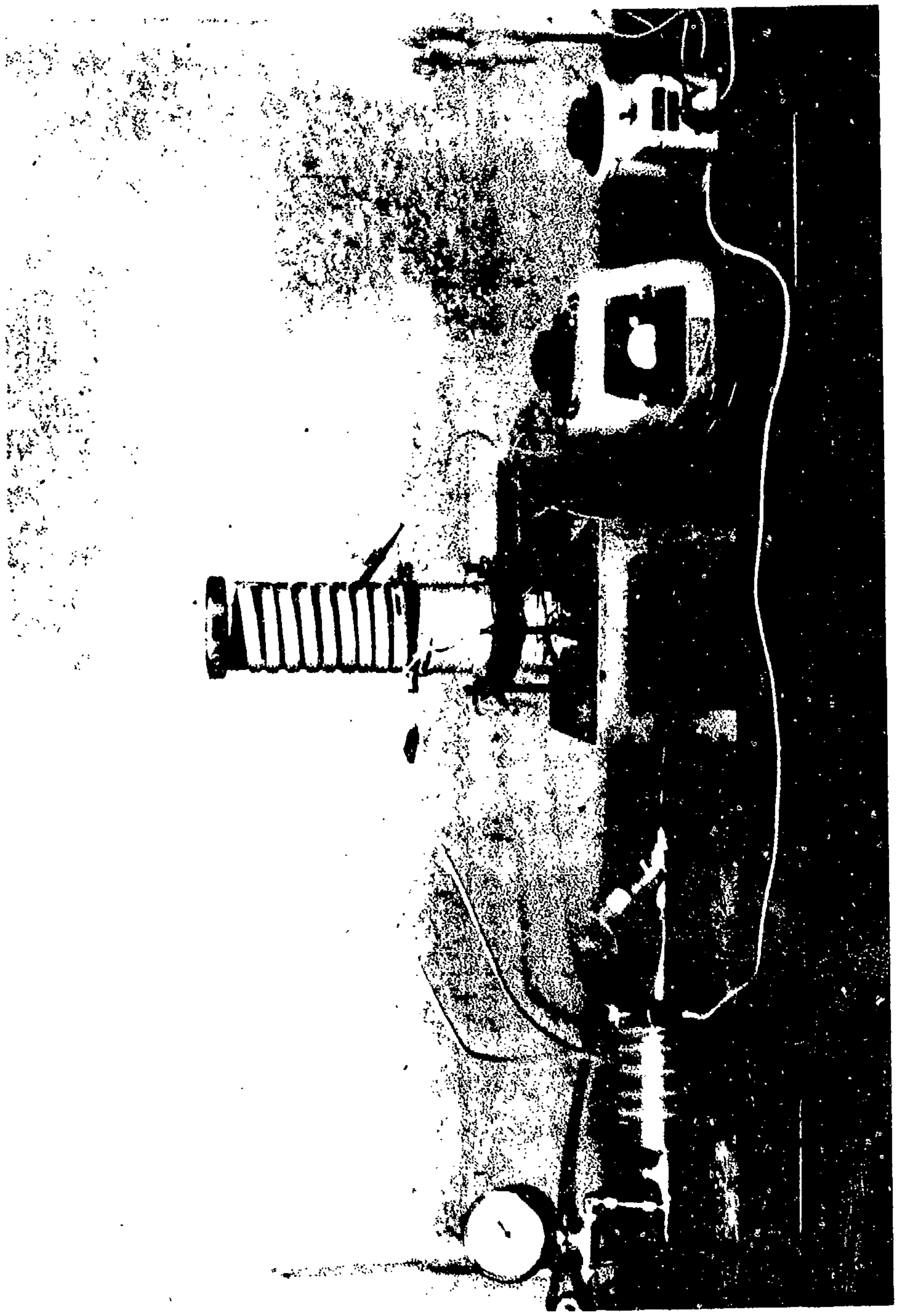

Figure 2. Hartmann apparatus.

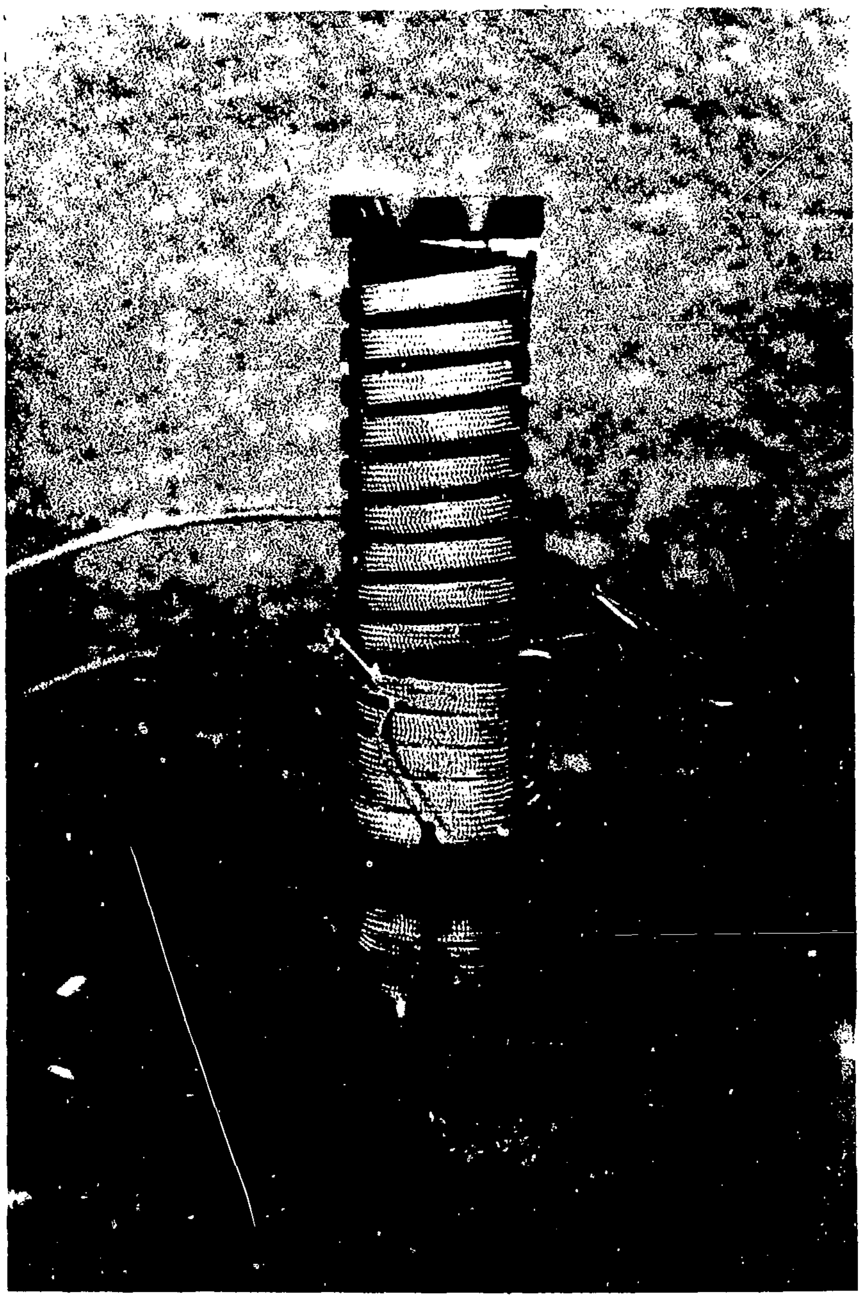

Figure 3. Heated Hartmann Lucite tube and dust dispersion cup.

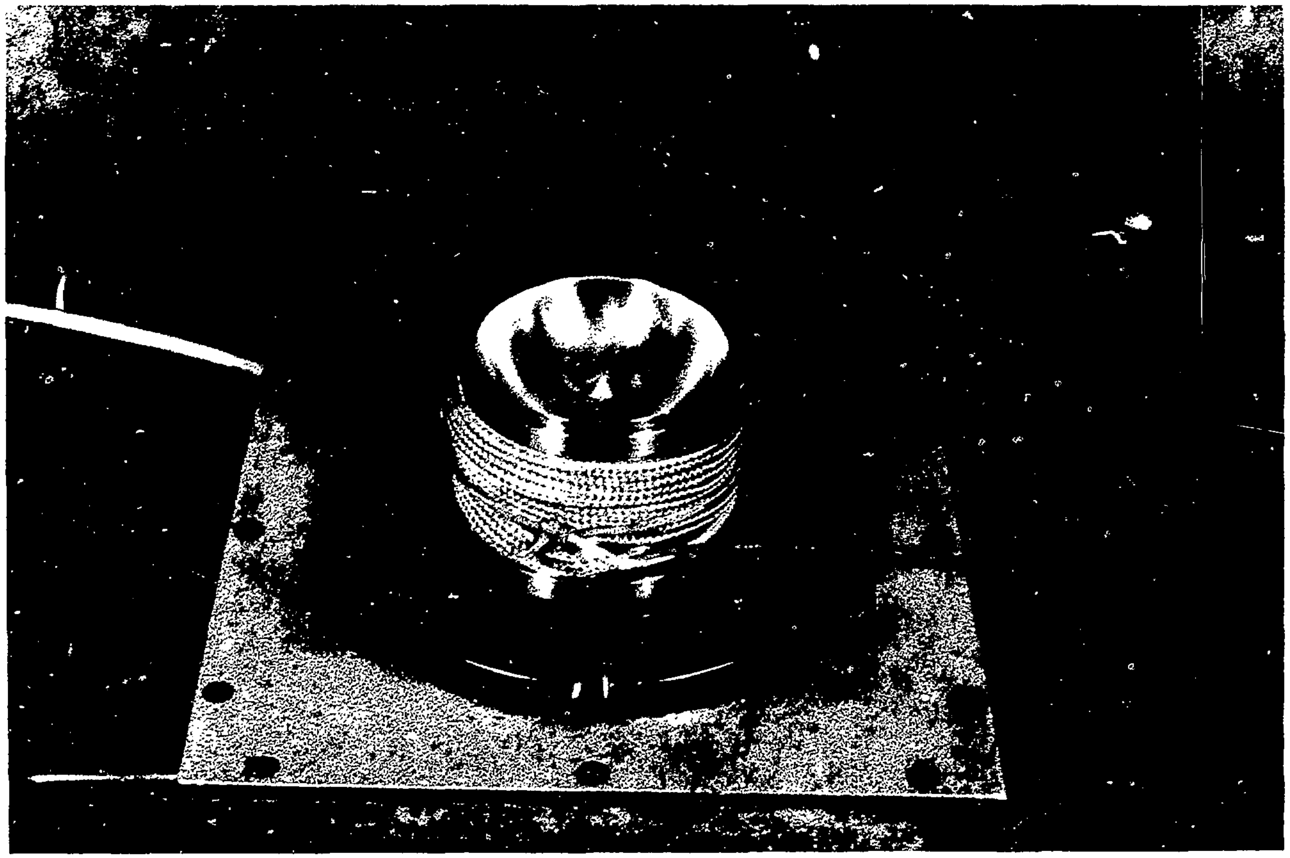

Figure 4. Dust dispersion cup.

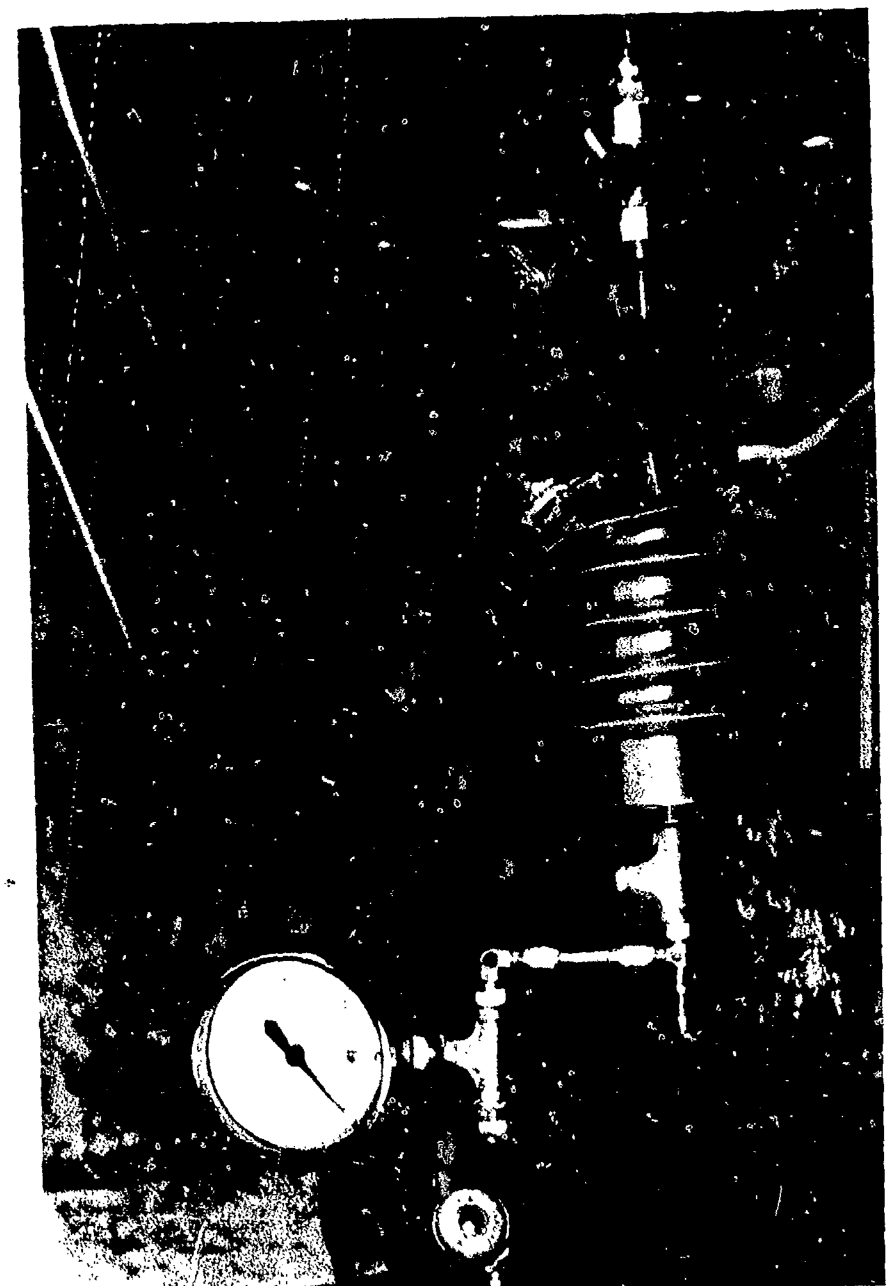

Figure 5. Air reservoir for dust dispersion.

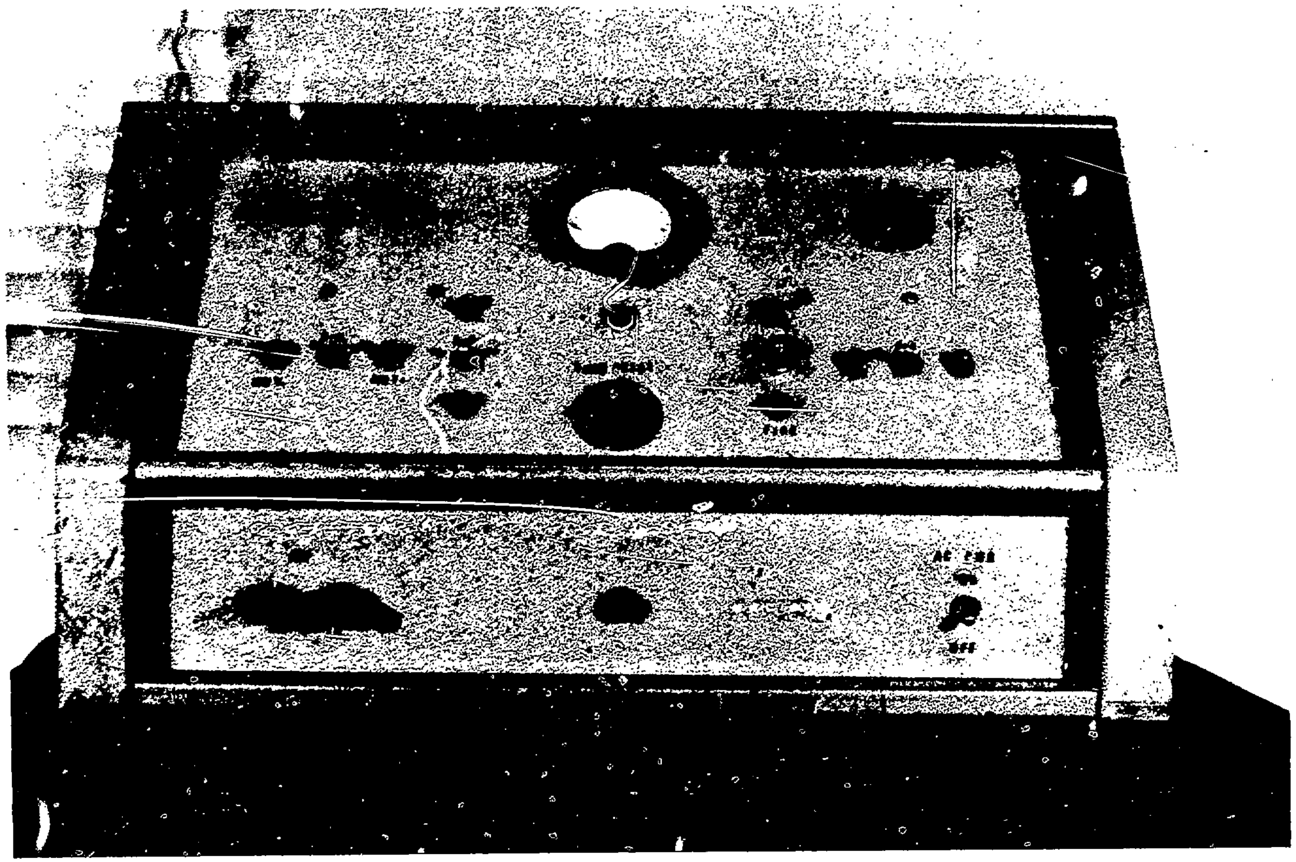

Figure 6. Electronic control console.

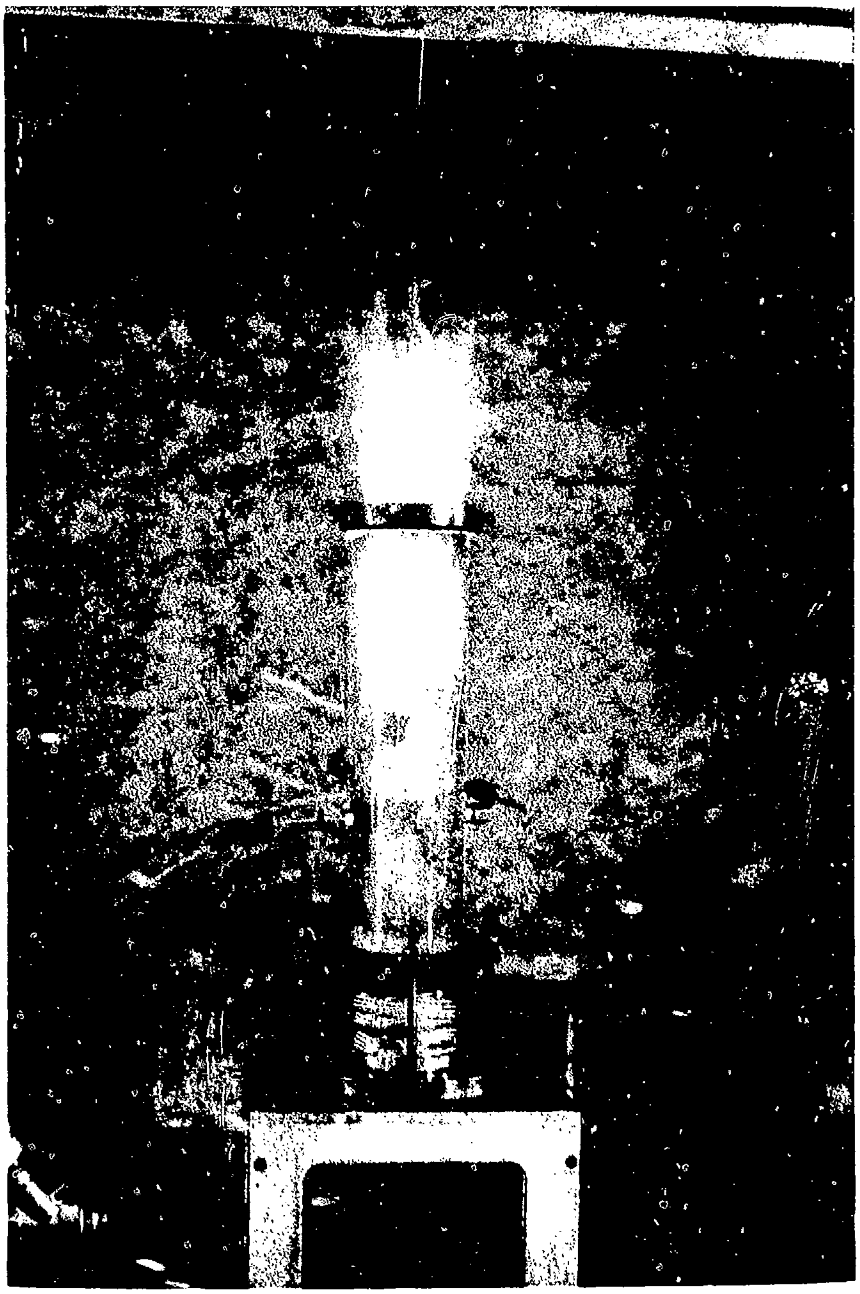

Figure 7. Dust explosion in Lucite tube.

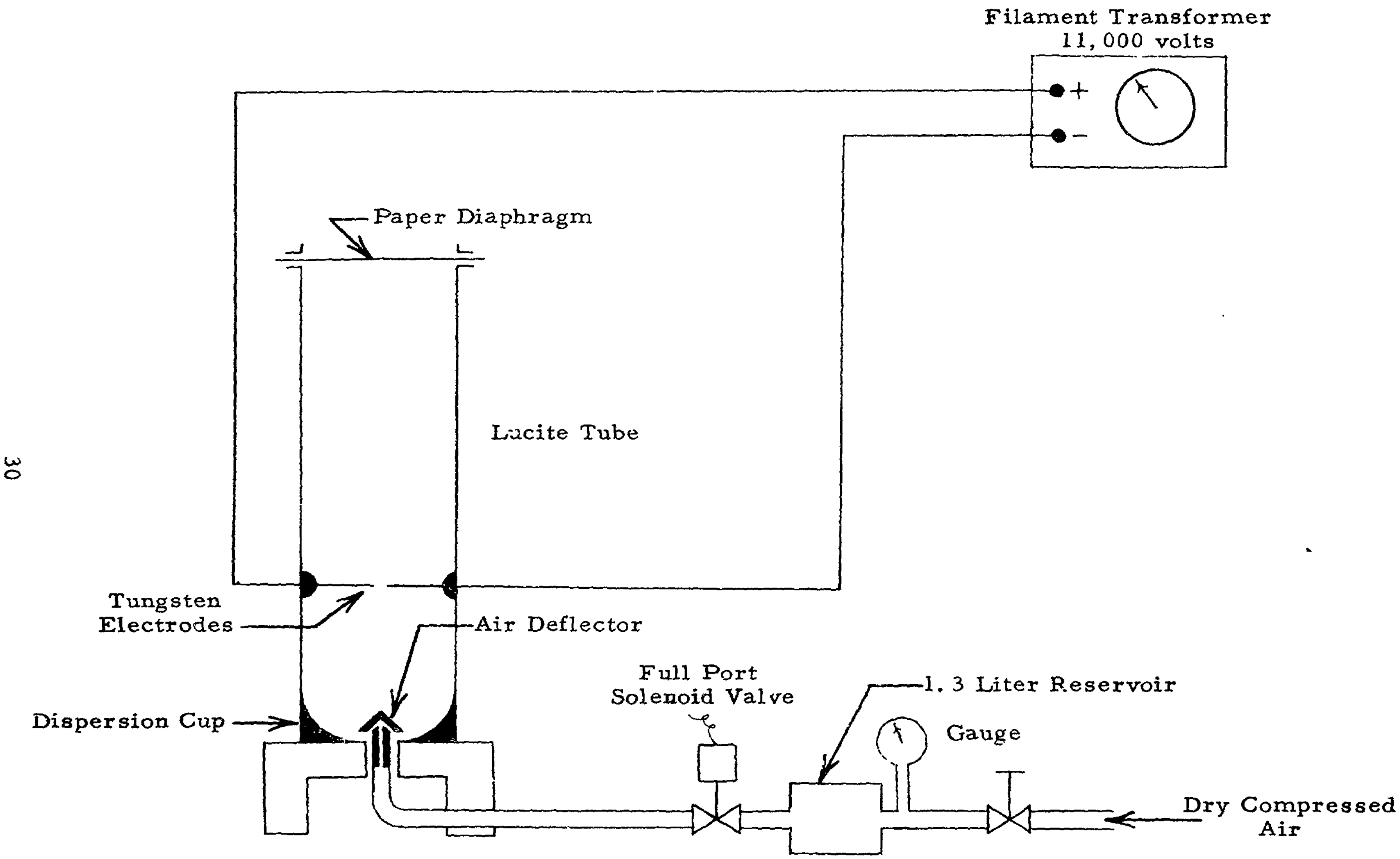


Figure 8. Hartmann apparatus for determining minimum concentration for explosion of a dust cloud.

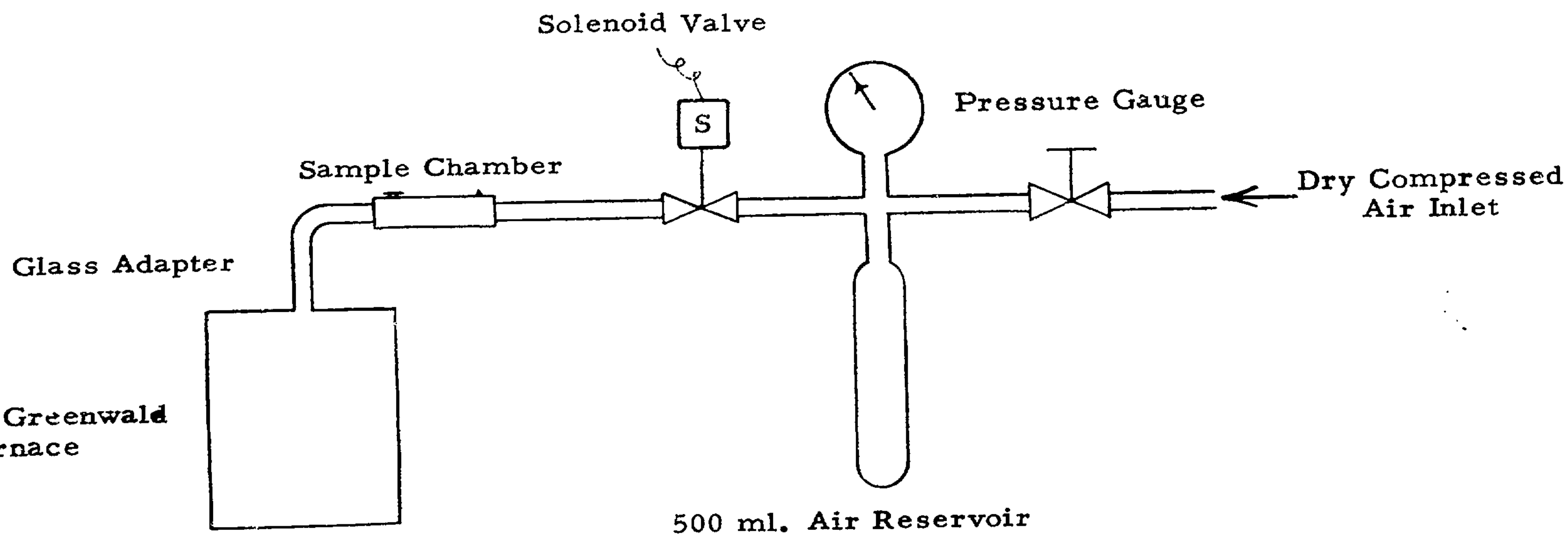


Figure 9. Schematic of minimum ignition temperature of dust cloud apparatus.

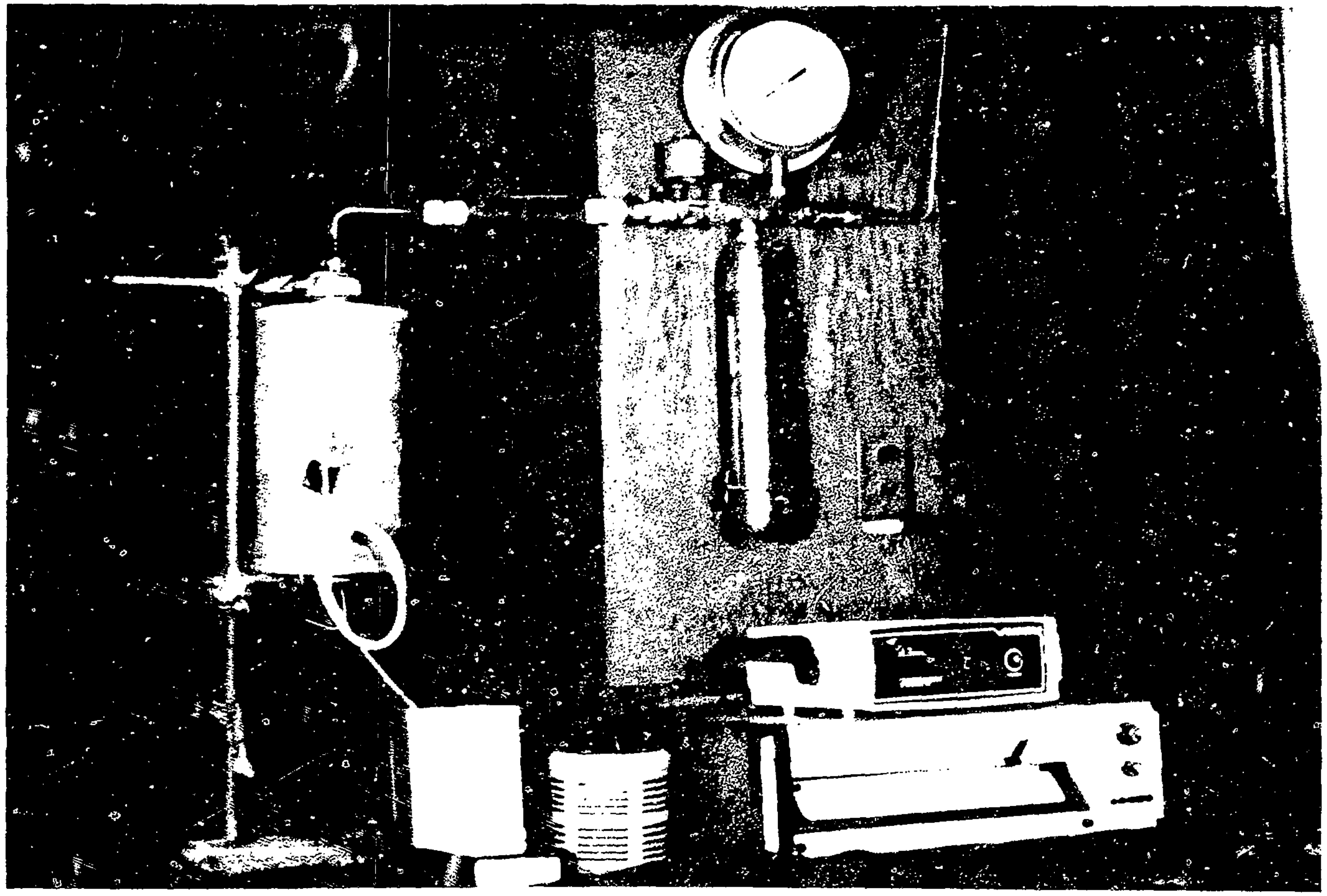

Figure 10. Minimum ignition temperature of a dust cloud apparatus.

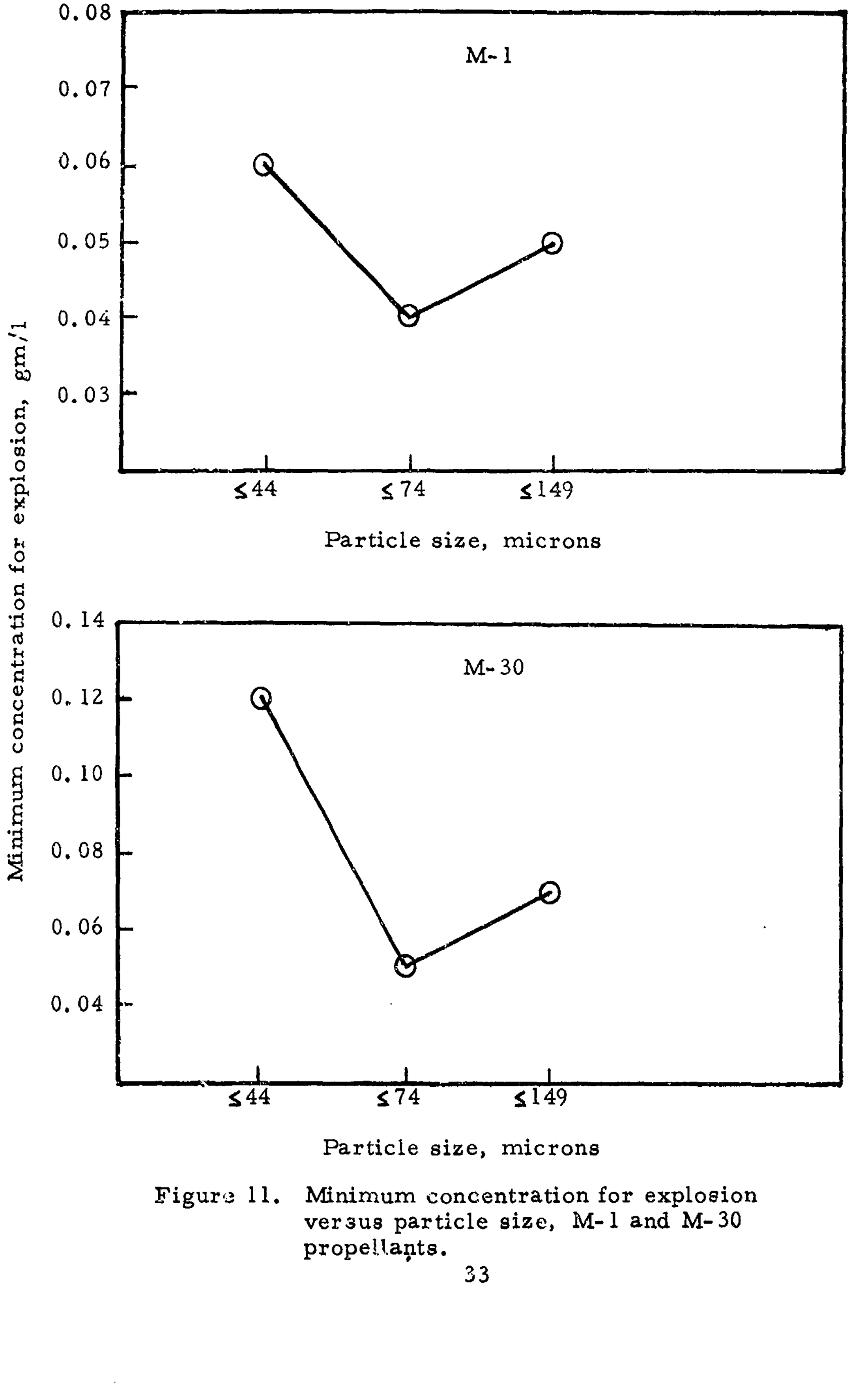


Figure 11. Minimum concentration for explosion versus particle size, M-1 and M-30 propellants.

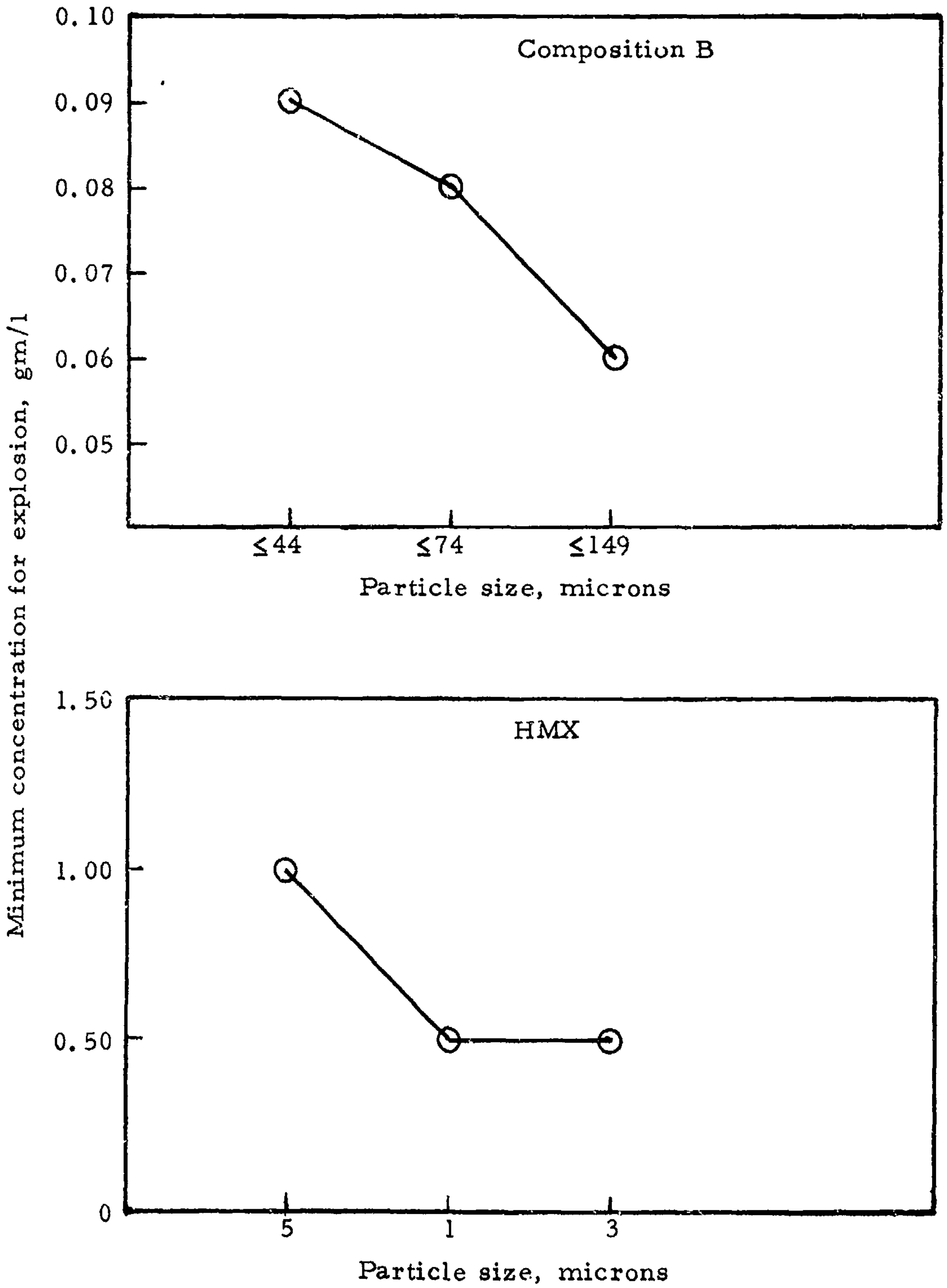


Figure 12. Minimum concentration for explosion versus particle size, Composition B and HMX explosives.

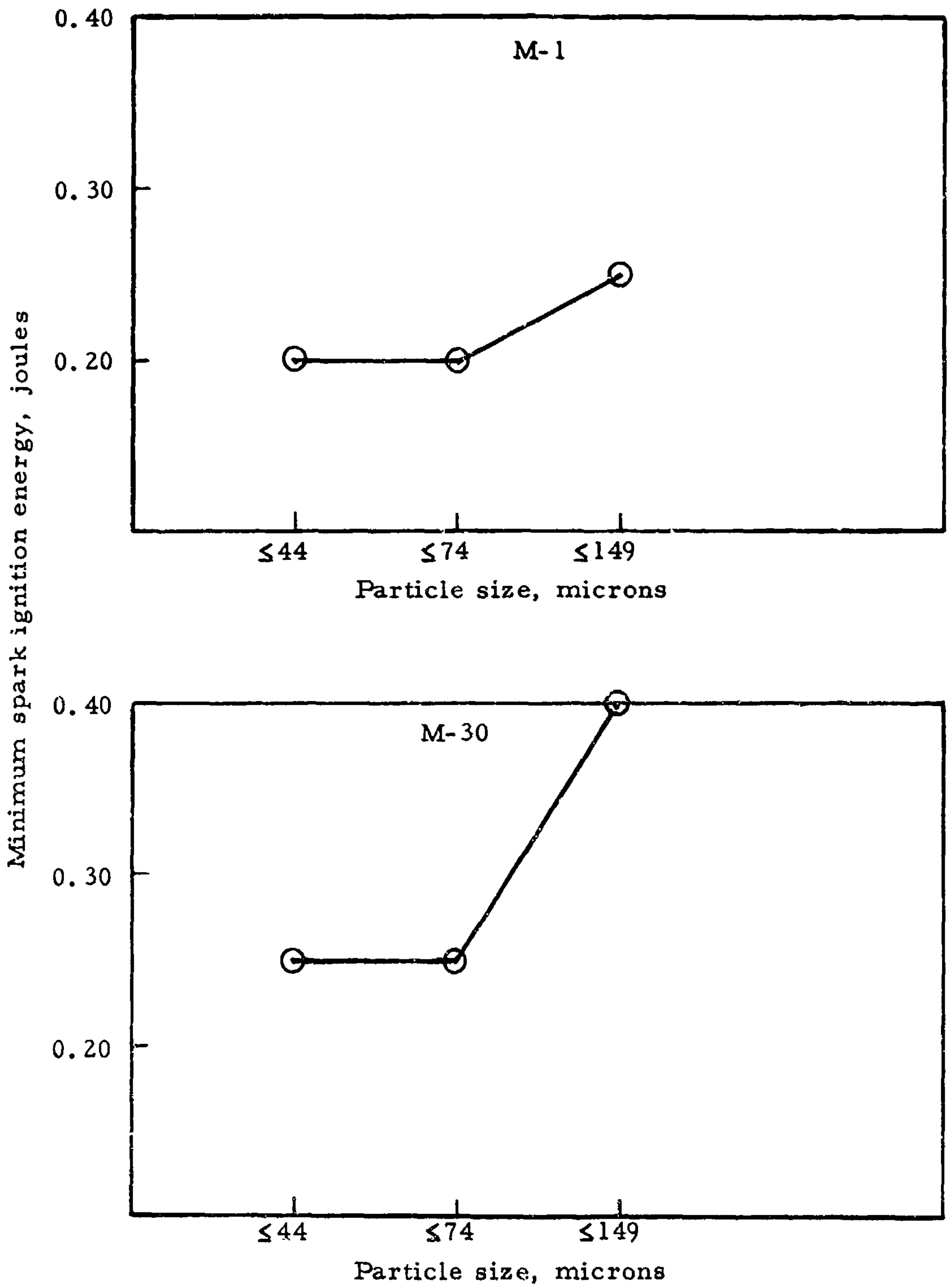


Figure 13. Minimum spark ignition energy versus particle size, M-1 and M-30 propellants.

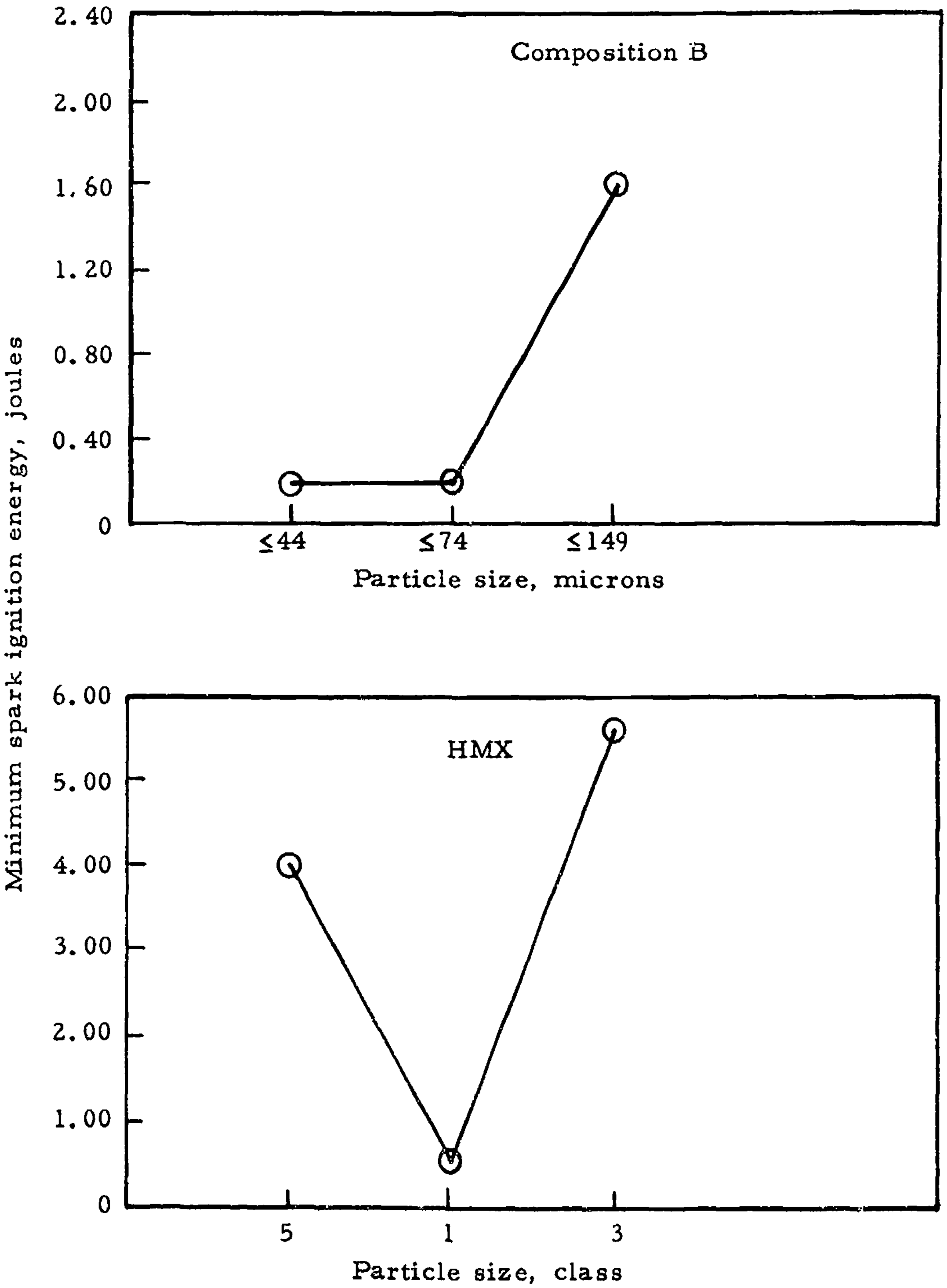


Figure 14. Minimum spark ignition energy versus particle size, Composition B and HMX explosives.

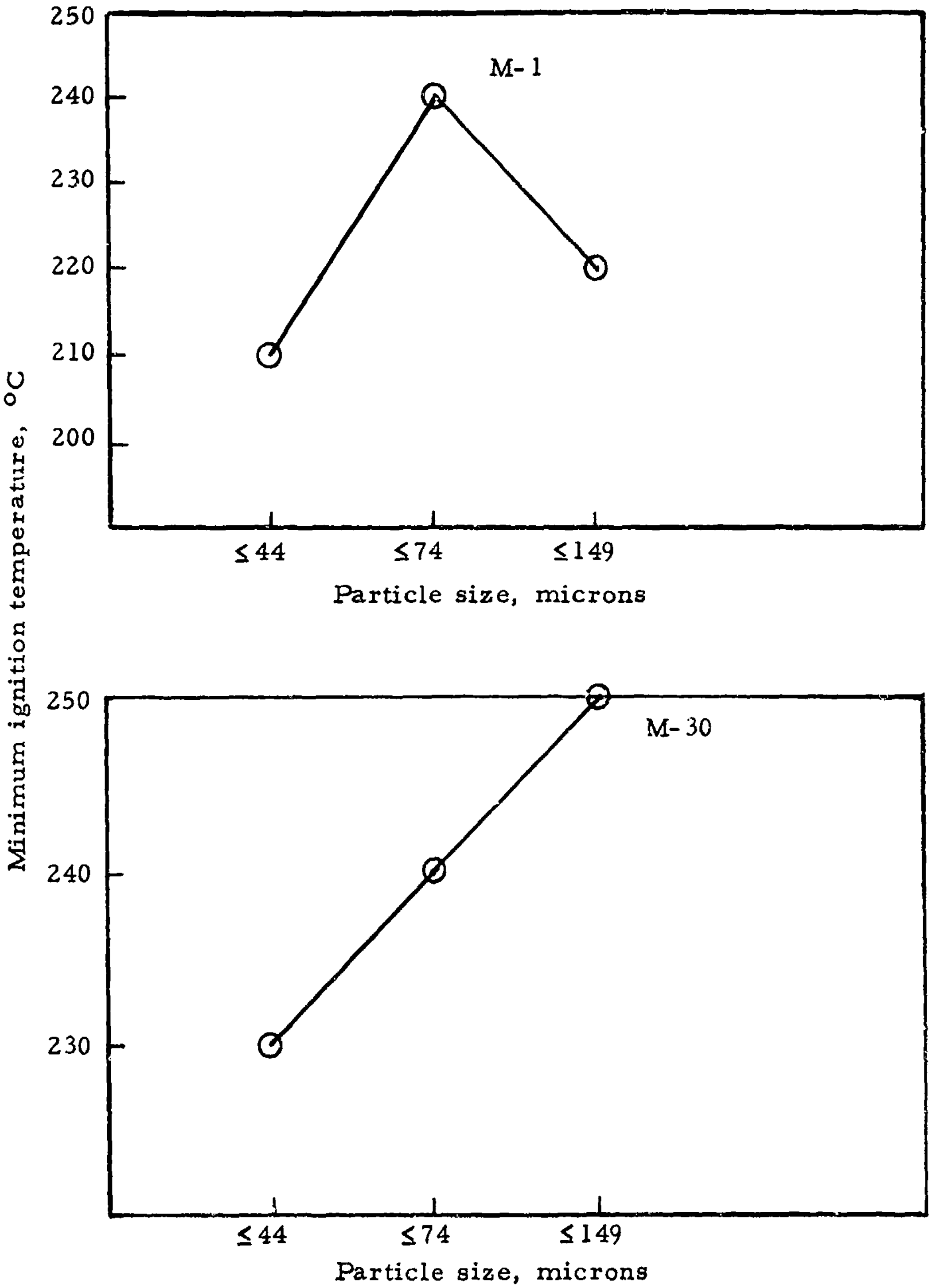


Figure 15. Minimum ignition temperature of dust clouds versus particle size, M-1 and M-30 propellants.

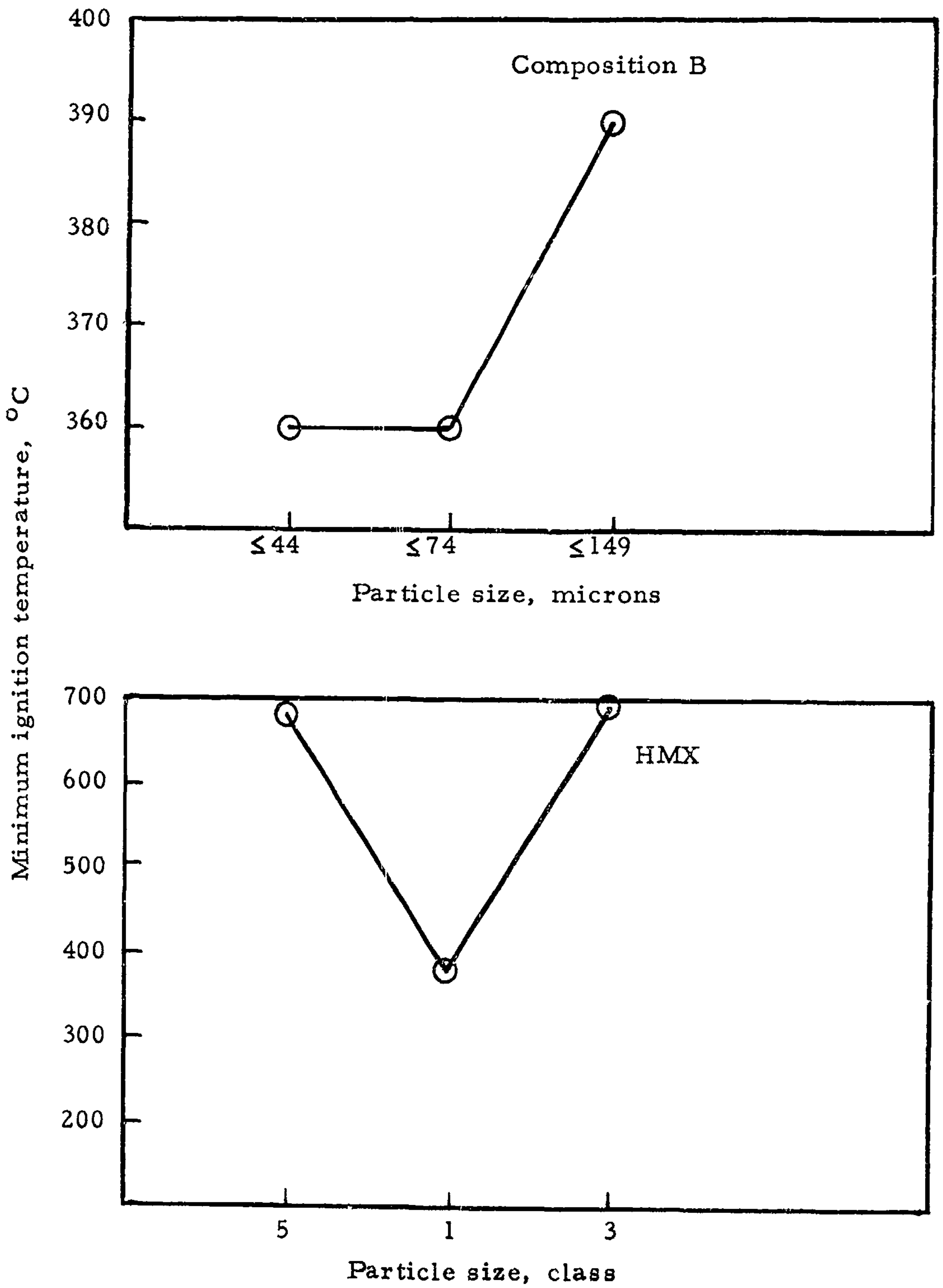


Figure 16. Minimum ignition temperature of dust clouds versus particle size, Composition B and HMX explosives.

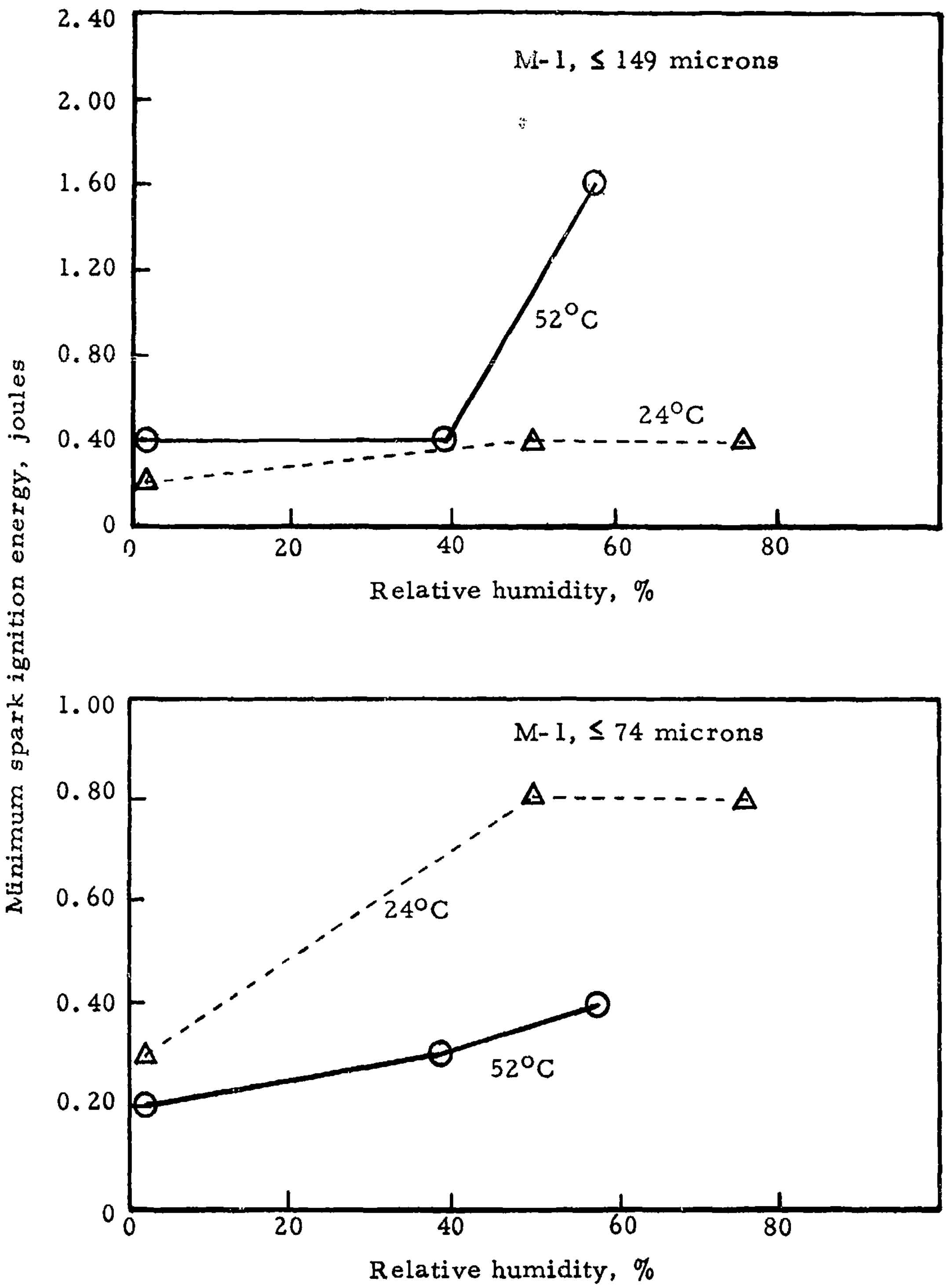


Figure 17. Minimum spark ignition energy versus relative humidity, M-1 propellant, particle size ≤ 149 and ≤ 74 microns.

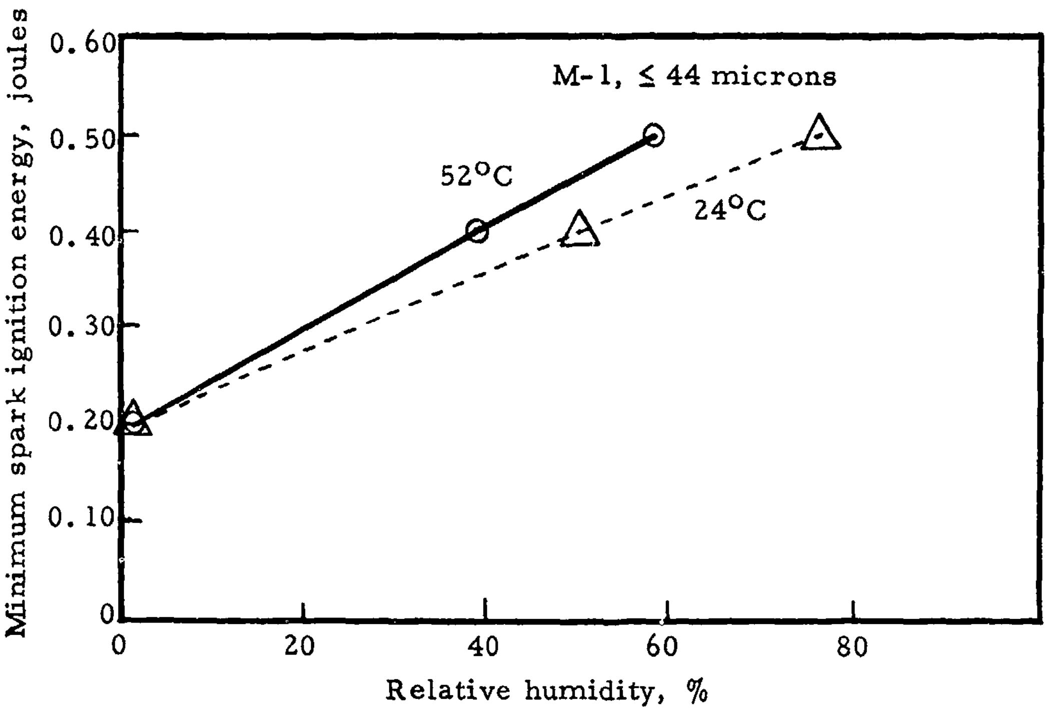


Figure 18. Minimum spark ignition energy versus relative humidity, M-1 propellant, particle size ≤ 44 microns.

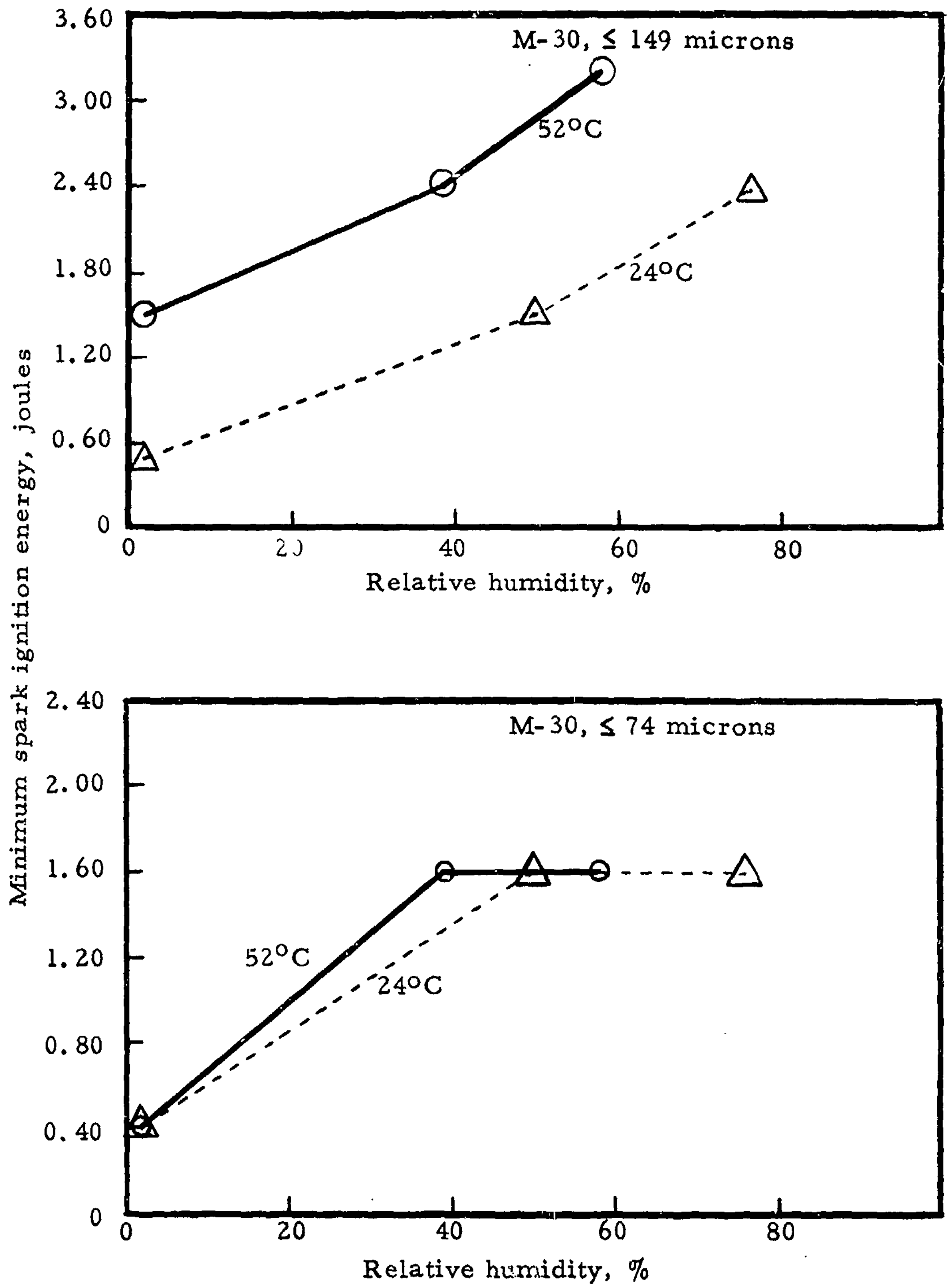


Figure 19. Minimum spark ignition energy versus relative humidity, M-30 propellant, particle size ≤ 149 and ≤ 74 microns.

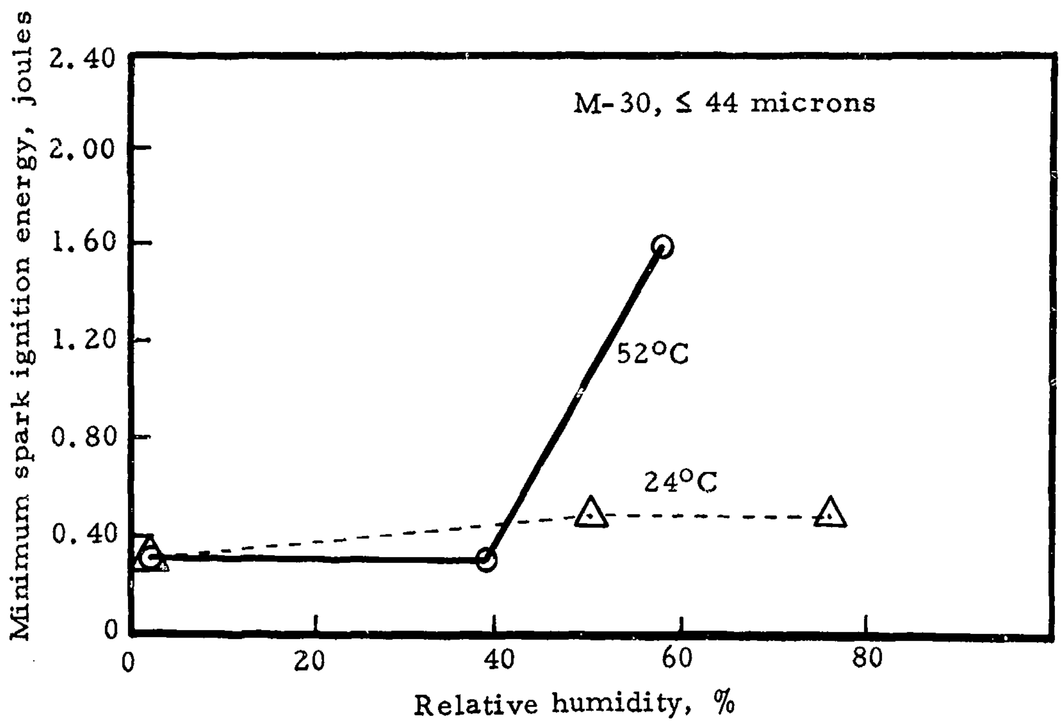


Figure 20. Minimum spark ignition energy versus relative humidity, M-30 propellant, particle size ≤ 44 microns.

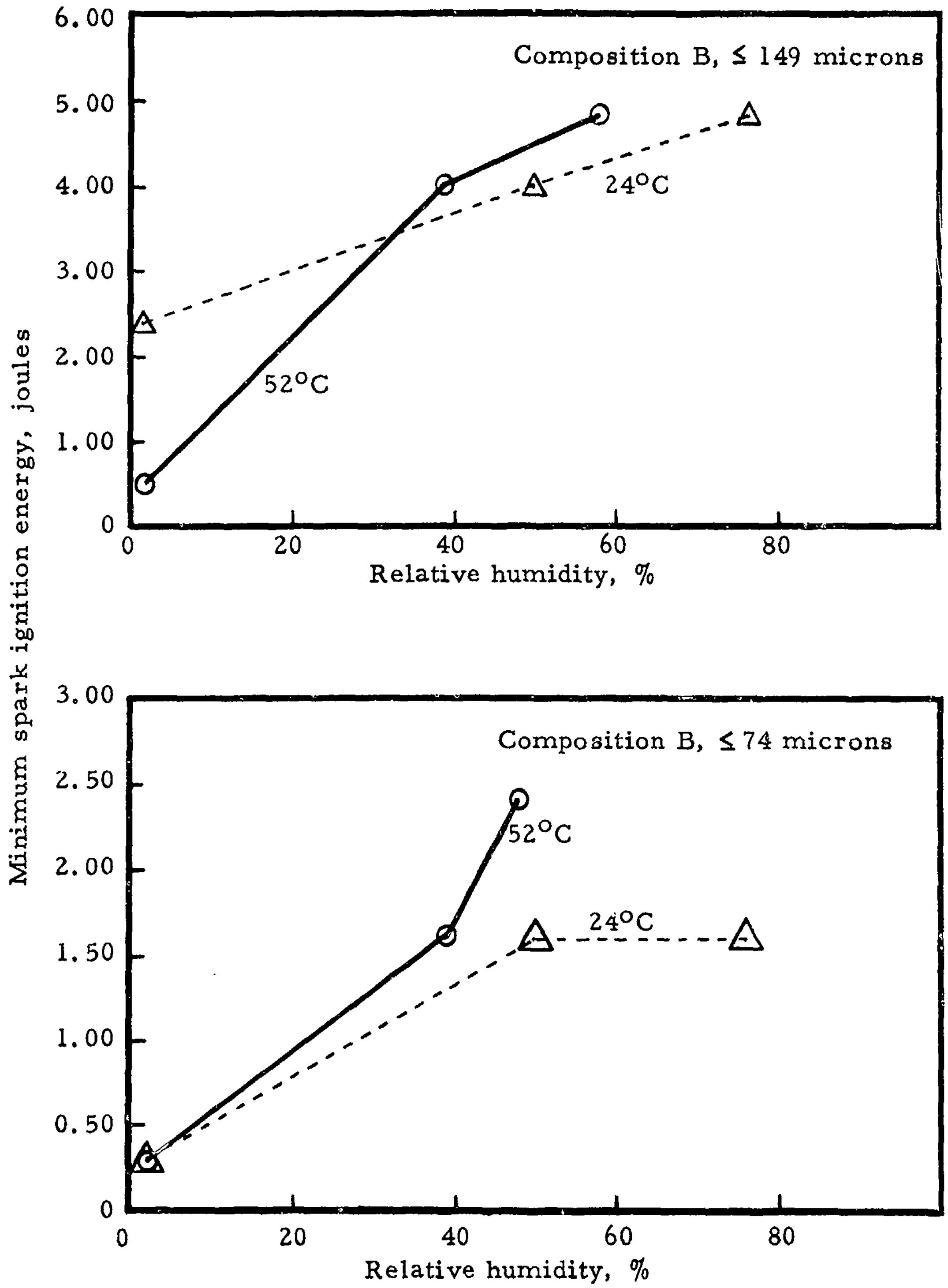


Figure 21. Minimum spark ignition energy versus relative humidity, Composition B explosive, particle size ≤ 149 and ≤ 74 microns.

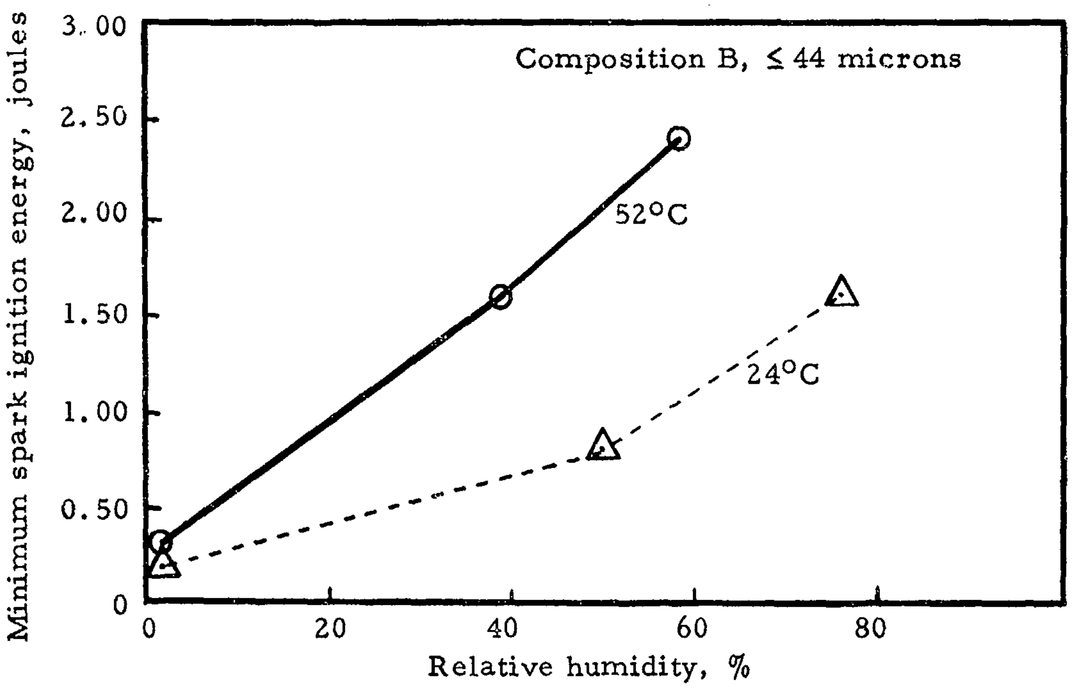


Figure 22. Minimum spark ignition energy versus relative humidity, Composition B explosive, particle size ≤ 44 microns.

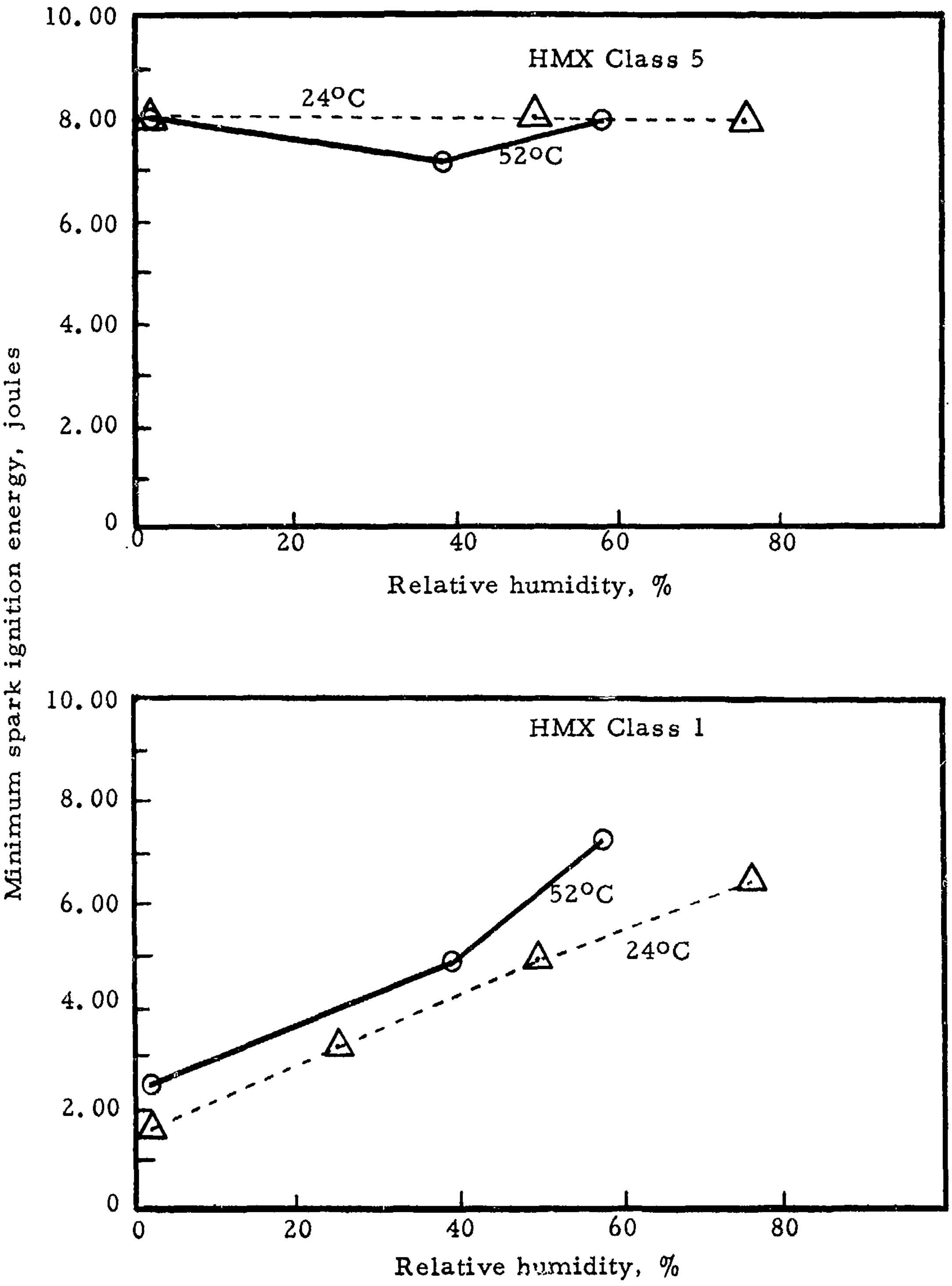


Figure 23. Minimum spark ignition energy versus relative humidity, HMX explosive, particle size class 5 and class 1.

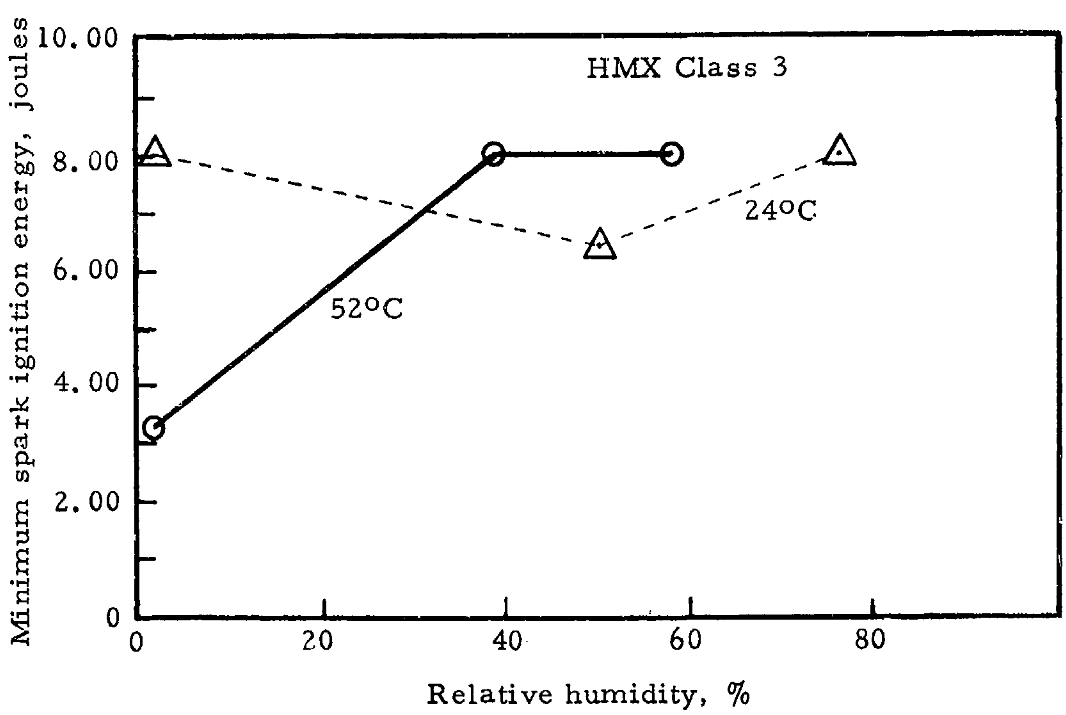


Figure 24. Minimum spark ignition energy versus relative humidity, HMX explosive, particle size class 3.

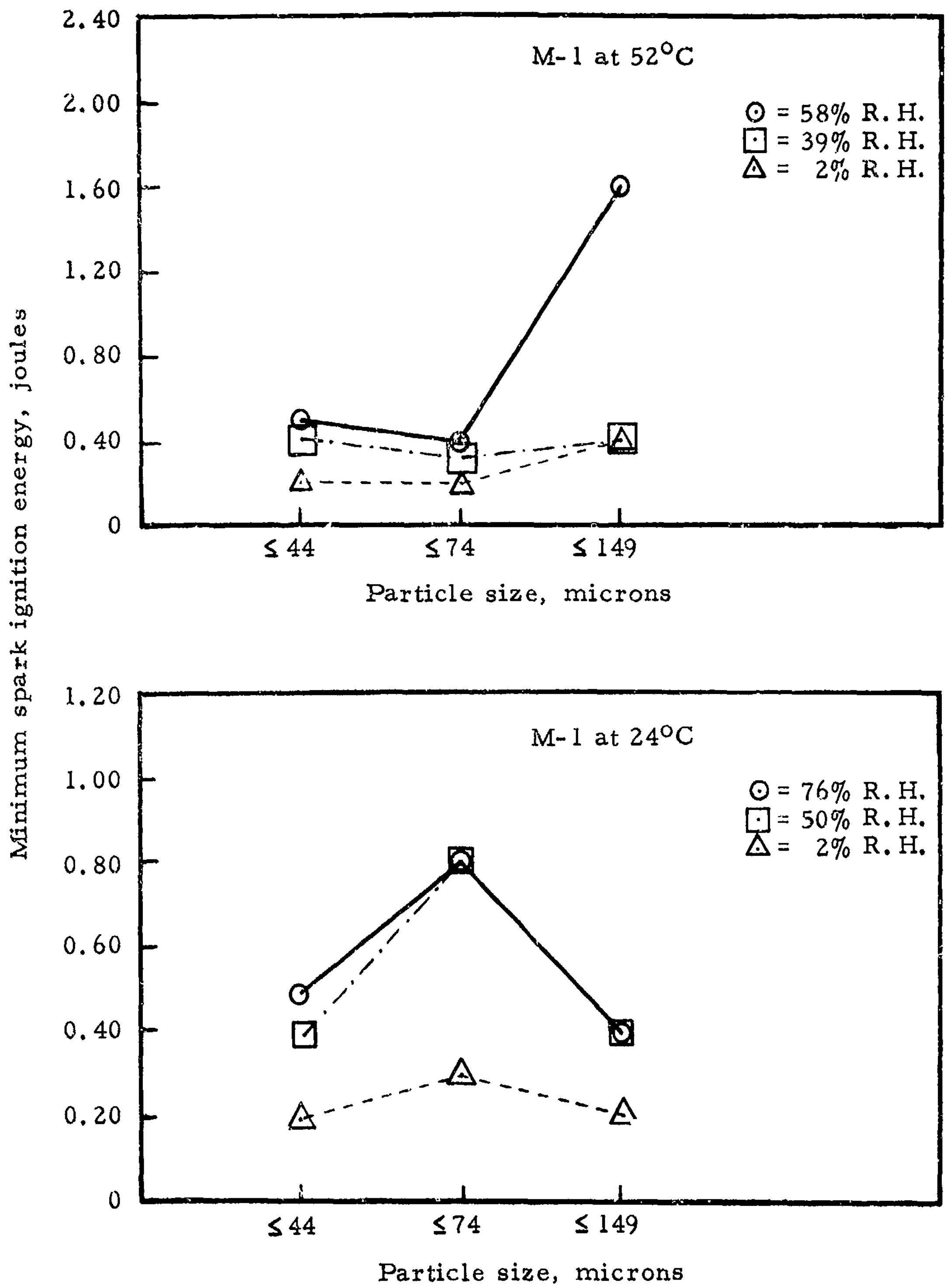


Figure 25. Minimum spark ignition energy versus particle size, M-1 propellant.

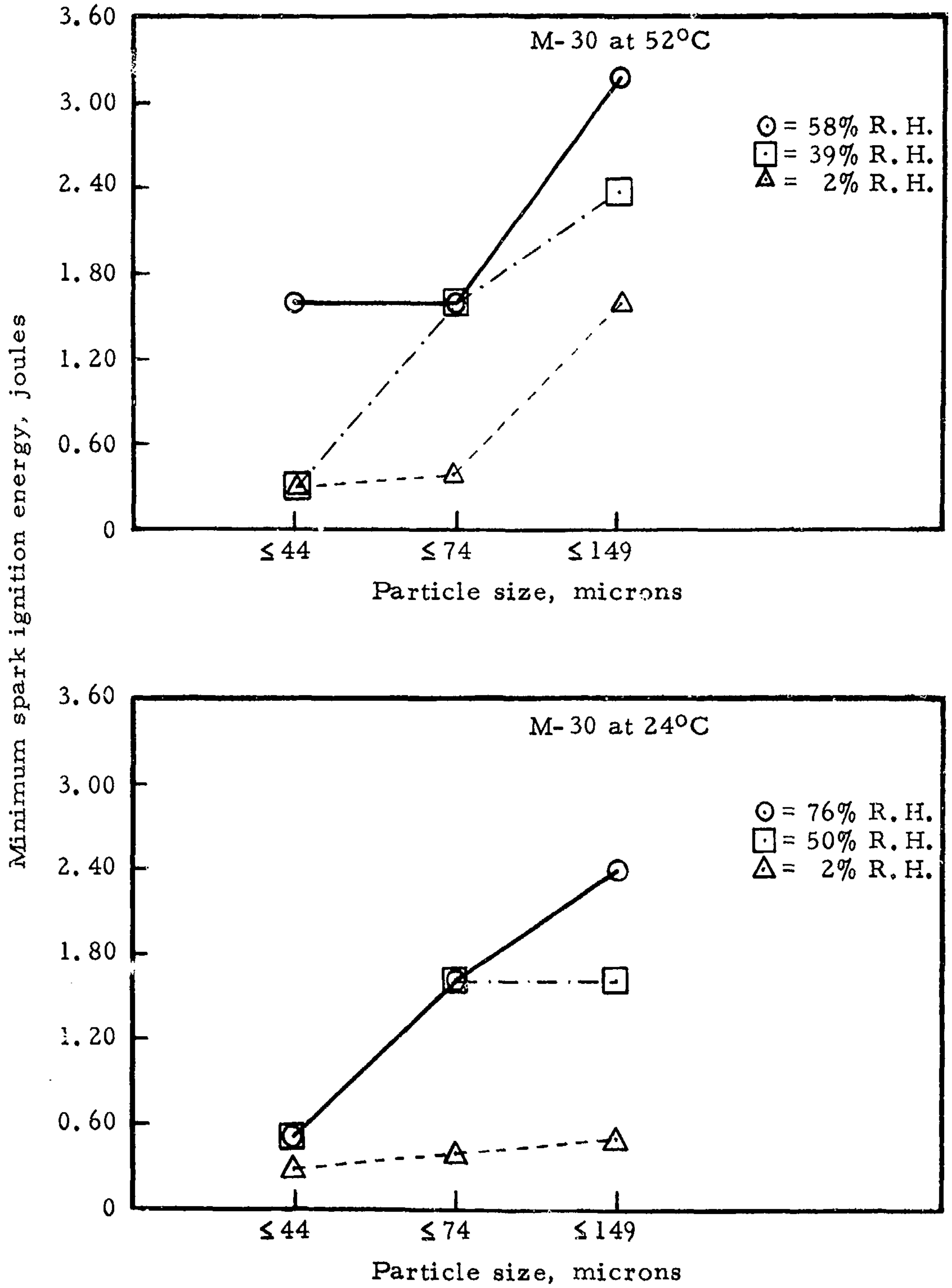


Figure 26. Minimum spark ignition energy versus particle size, M-30 propellant.

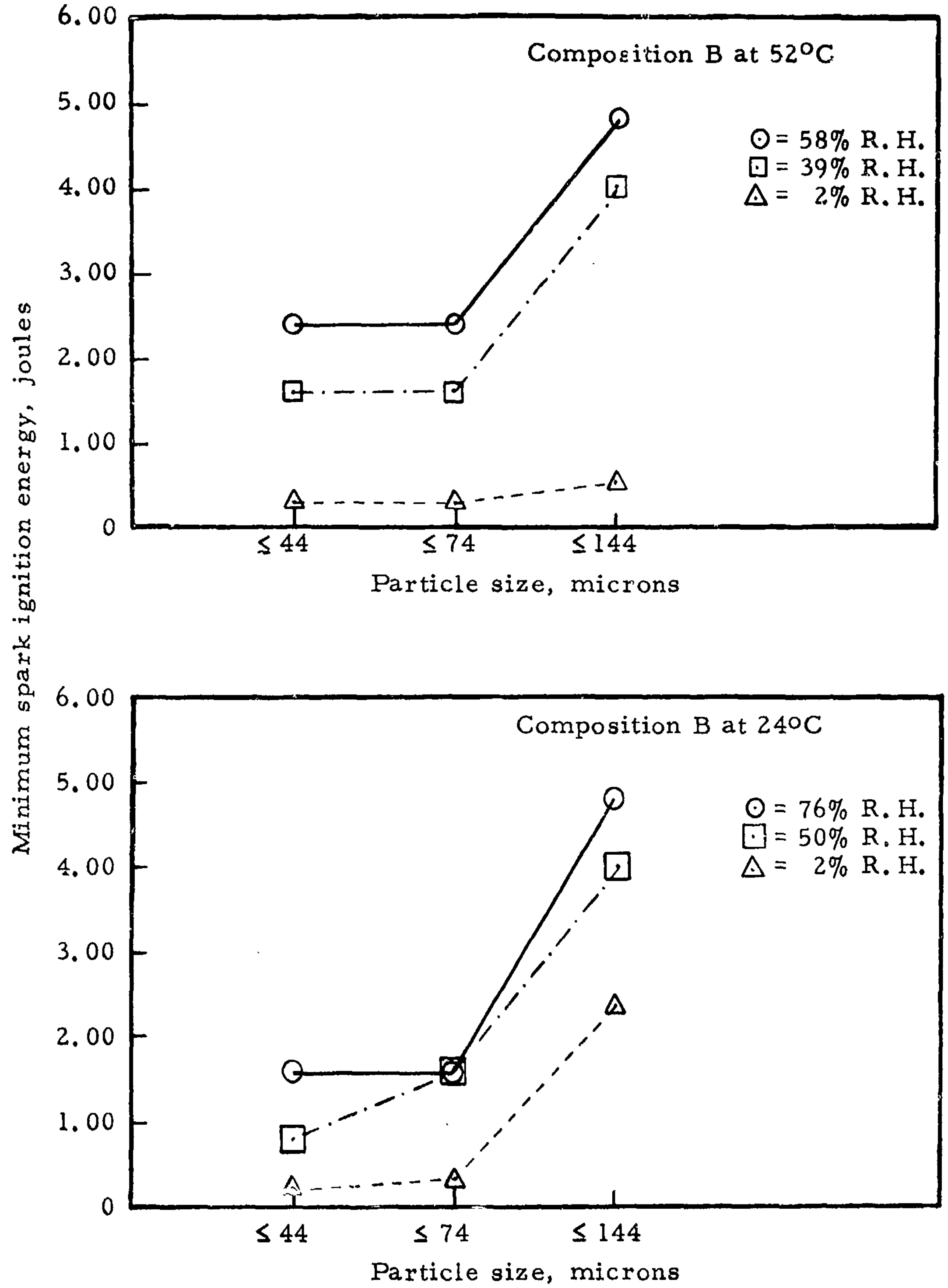


Figure 27. Minimum spark ignition energy versus particle size, Composition B explosive.

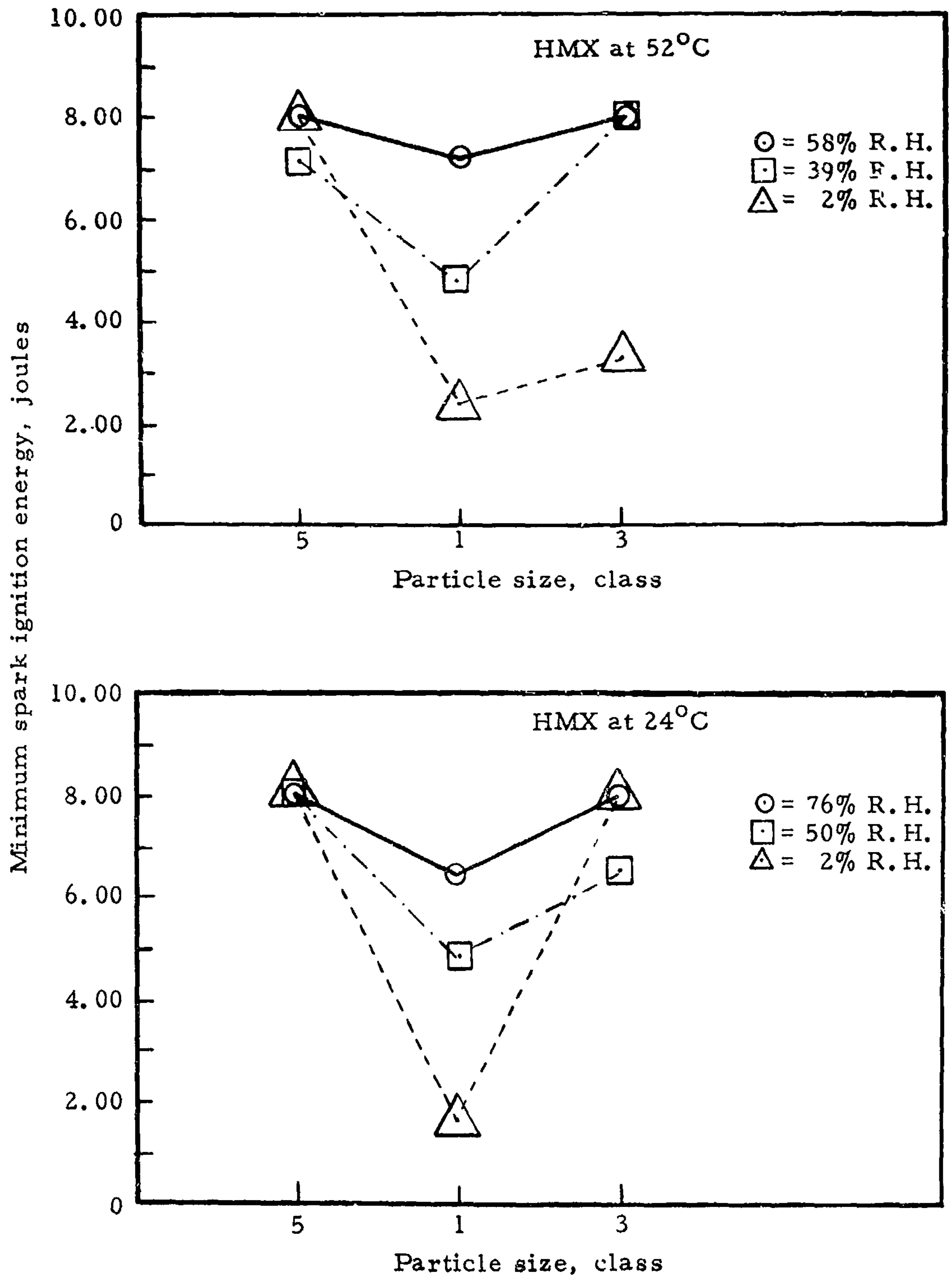


Figure 28, Minimum spark ignition energy versus particle size, HMX explosive.

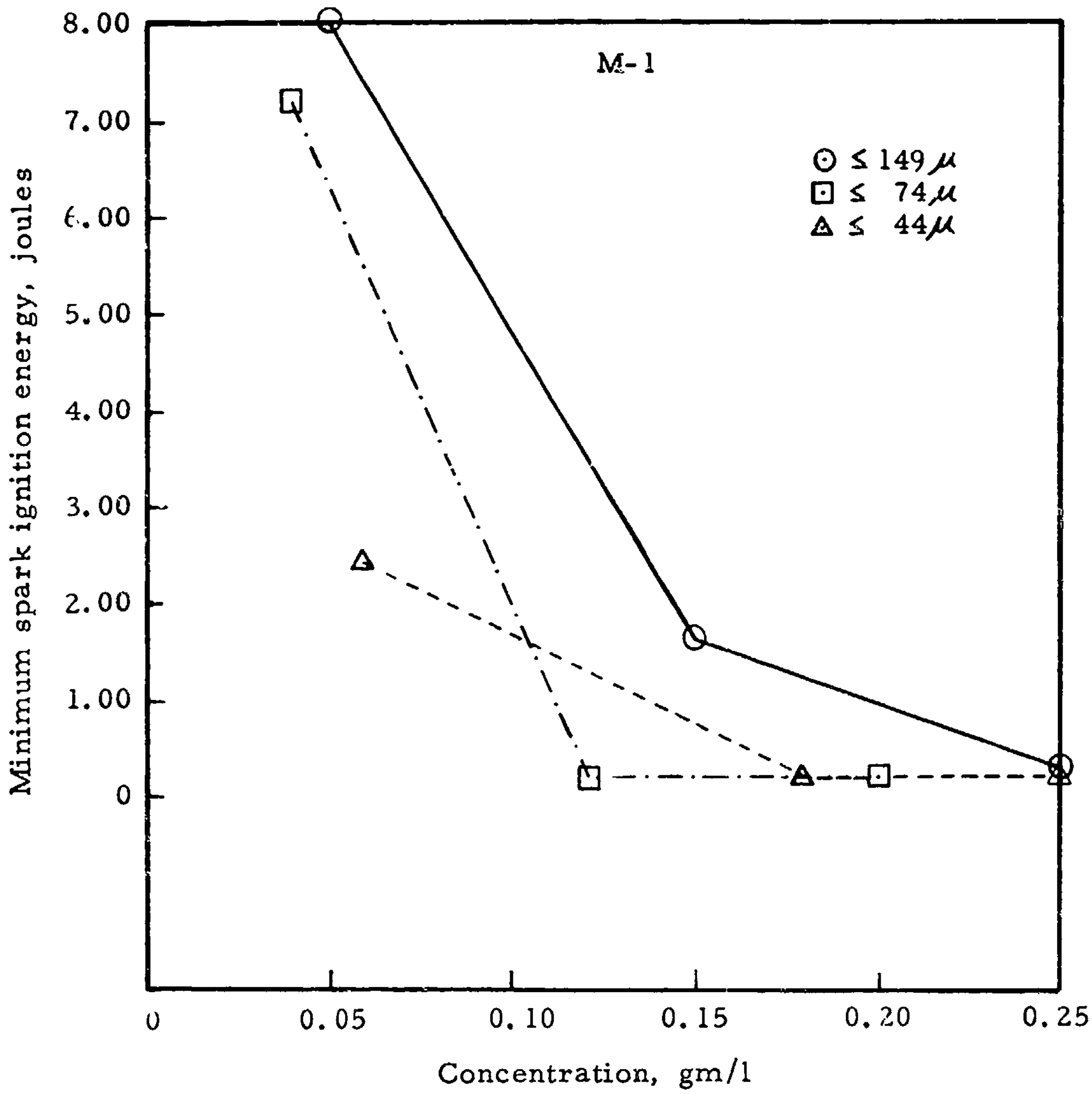


Figure 29. Minimum spark ignition energy versus concentration, M-1 propellant.

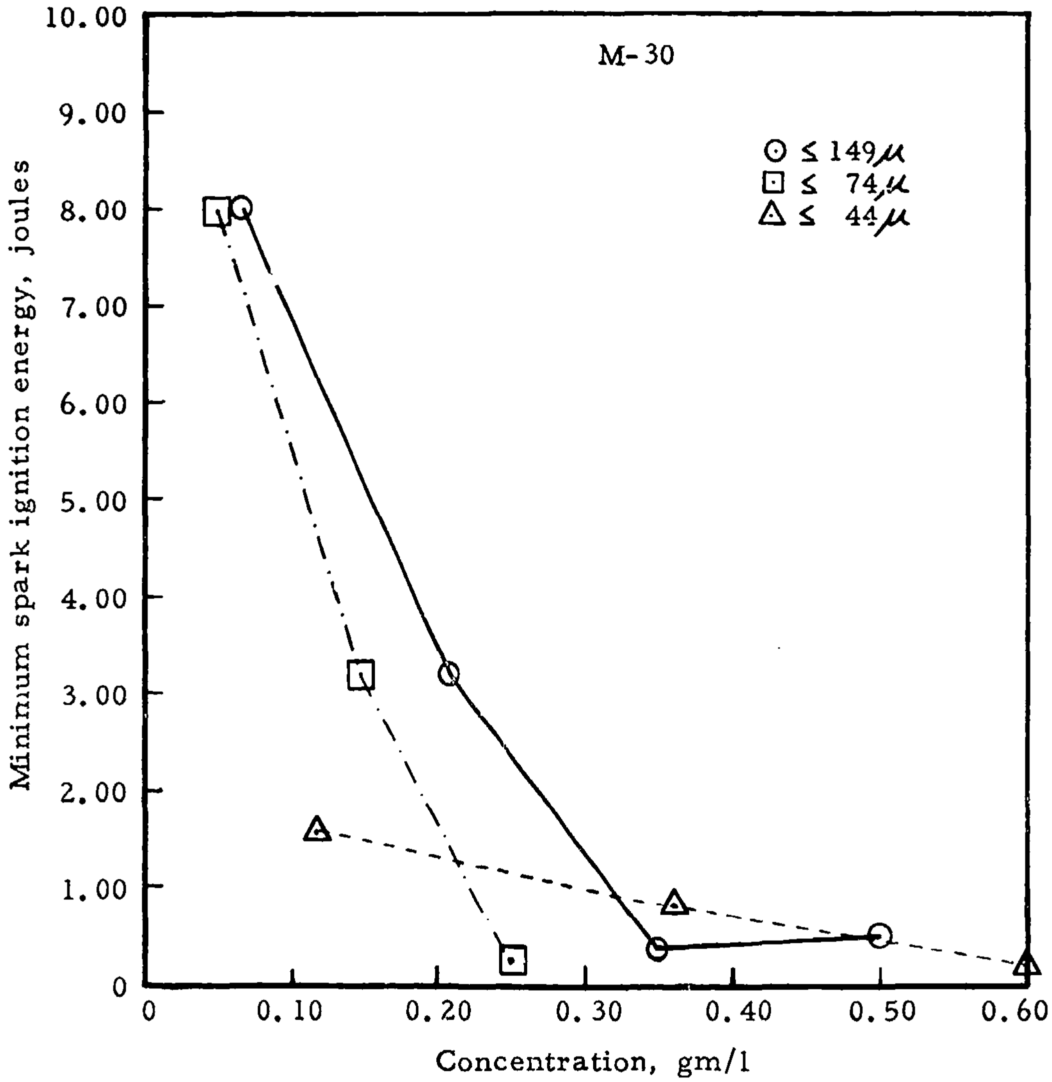


Figure 30. Minimum spark ignition energy versus concentration, M-30 propellant.

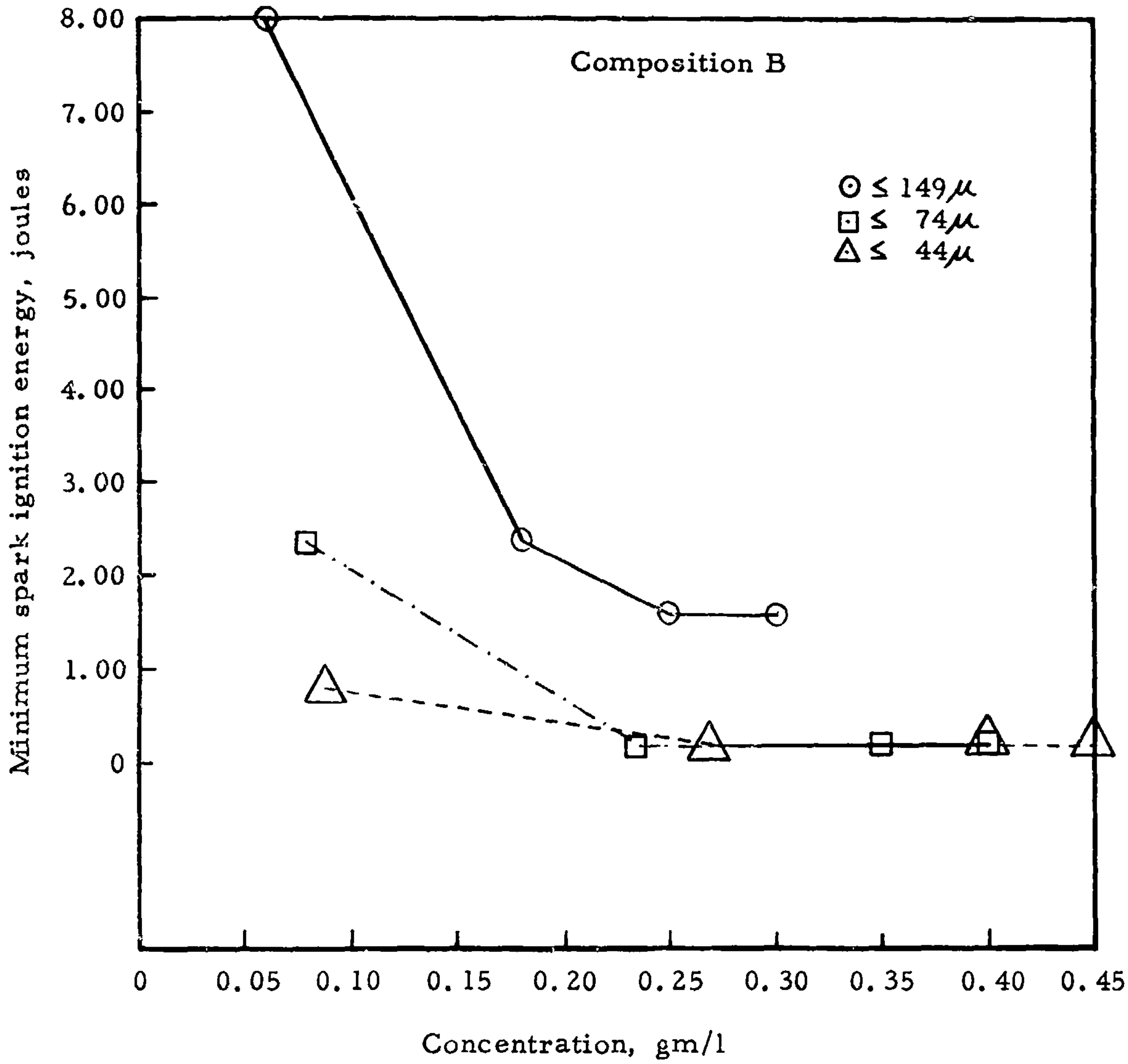


Figure 31. Minimum spark ignition energy versus concentration, Composition B explosive.

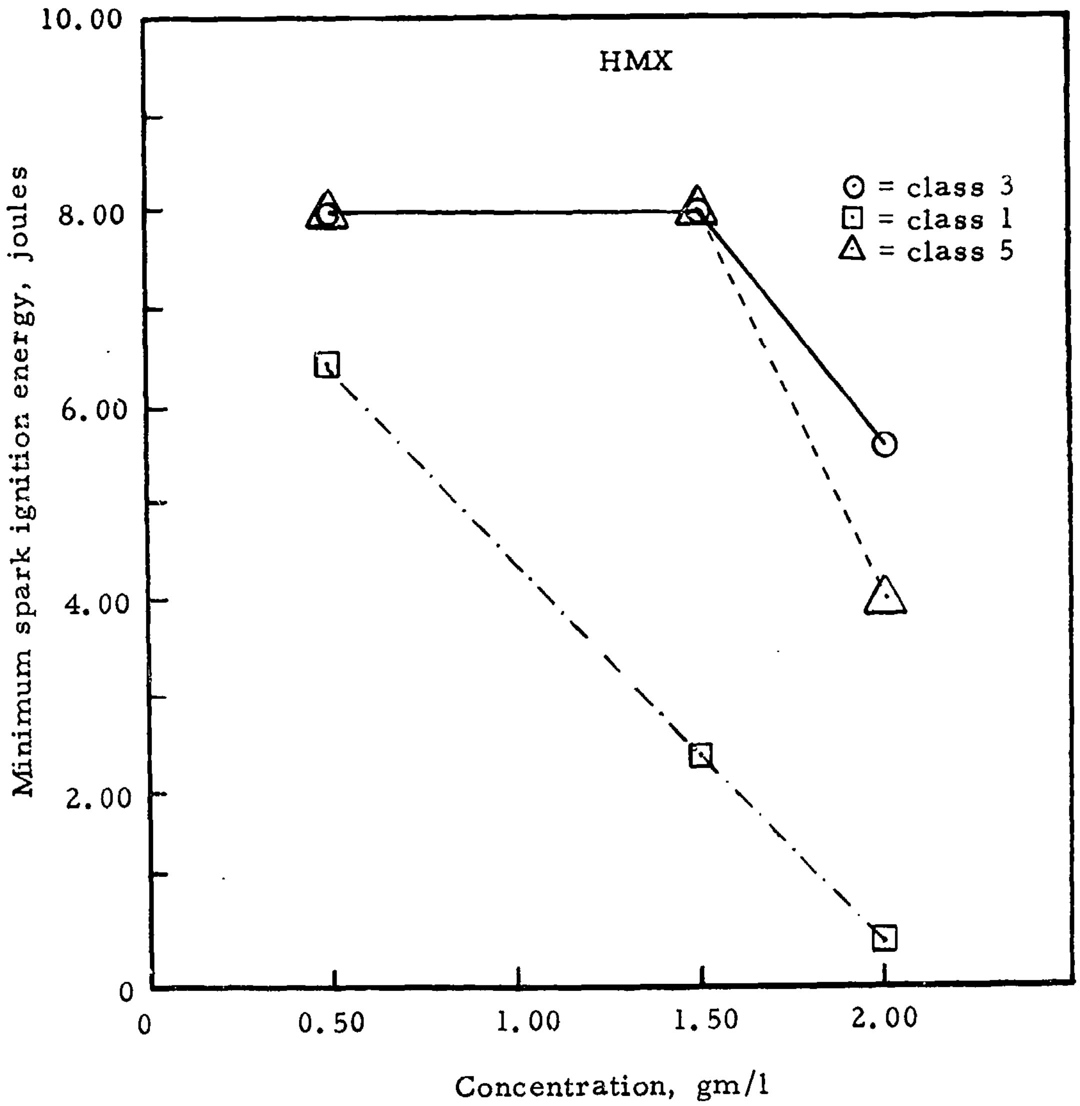


Figure 32. Minimum spark ignition energy versus concentration, HMX explosive.

# APPENDIX A

# MATERIALS AND EQUIPMENT

## MATERIALS AND EQUIPMENT

### Materials

The following materials were supplied by ARRADCOM for use in this test program:

(1) HMX, Grade B, Classes 1, 3 and 5, Lots GHBC-4, 6HG5-3, 6HCC-11A2

(2) Composition B, Grade A, Spec. Mil C 0040 A, Lot HOL-053-5068 (screened)

(3) M-30 Propellant, Mil P-46458, Lot RAD 67595 (Class 2)

(4) M-1 Propellant

### Equipment

ARRADCOM supplied the following equipment:

(1) Aqua Test II, Model #702 by Photo Volt Corporation, S/N 1095 (automatic Karl Fischer titrator)

Hazards Research Corporation supplied the following equipment:

(1) Hartmann Explosibility Apparatus

(2) Godbert-Greenwald Furnace

(3) Moulinex grinder

(4) Tyler Sieve Shaker

(5) U. S. Sieves, No. 100 (149 micron opening), No. 200 (74 micron opening), No. 325 (44 micron opening)

# APPENDIX B

## DESCRIPTION OF EXPERIMENTAL METHODS

## Sample Preparation

Composition B, M-1 and M-30 samples that satisfied the through 100, 200 and 325 U.S. sieve size requirement were not readily available for use on this program. Consequently, HRC prepared the samples by remotely impact grinding the dry materials. The samples were prepared in 20 gram batches. Each batch was made by subjecting the material to the grinding action for between 20 and 60 seconds. Duration was the controlling parameter for particle size reduction. Final particle size classification was accomplished by sieving the sample through the appropriate U.S. series sieve. All samples were then stored in air tight containers for future testing.

HMX samples were tested as received from the Government. Three granulations were supplied, classes 1, 3 and 5. Military specification H-45444 B (PA) contains the particle size distribution for these three classes of HMX. This data is extracted from the specification and presented in table B-1 below:

Table B-1. HMX granulation specification

| Through U.S. standard sieve no. | Class 1 Percent | Class 3 Percent | Class 5 Percent |
|---|---|---|---|
| 12 | | 99 min | |
| 50 | $90 + 6$ | $40 + 15$ | |
| 100 | $50 \mp 10$ | $20 \mp 10$ | |
| 200 | $20 \mp 6$ | $10 \mp 10$ | |
| 325 | $8 \mp 5$ | | 98 min |

It is observed that the class 5 HMX contains the smallest particles followed by classes 1 and 3 in order of increasing particle size.

U.S. standard sieve numbers and corresponding sieve openings are presented in Table B-2 for future reference.

Table B-2. U.S. sieve openings

| Sieve designation (no.) | Sieve opening (microns) |
|---|---|
| 12 | 1680 |
| 50 | 297 |
| 100 | 149 |
| 200 | 74 |
| 325 | 44 |

## Sample Volatiles Content

The volatiles content of each material was determined by vacuum drying it for 6 hours at 60°C. Table B-3 presents the results of the volatiles content determinations.

Table B-3. Volatiles content of Comp B, HMX, M-1 and M-30 samples

| Sample | Particle size | Percent volatiles |
|---|---|---|
| Composition B | thru 100 mesh | 0.27 |
| | thru 200 mesh | 0.53 |
| | thru 325 mesh | 0.40 |
| HMX | Class 1 | 0.05 |
| | Class 3 | 0.02 |
| | Class 5 | 0.00 |
| M-1 | thru 100 mesh | 0.56 |
| | thru 200 mesh | 0.51 |
| | thru 325 mesh | 1.30 |
| M-30 | thru 100 mesh | 4.20 |
| | thru 200 mesh | 2.70 |
| | thru 325 mesh | 2.60 |

## Test Air Environmental Control Methods

In order to meet the objectives of Phase 2, it was necessary to develop a technique for controlling the temperature and relative humidity of the air in the Hartmann apparatus. The goal was to subject each dust cloud to a specific environmental condition and to determine the effect this condition had on the minimum ignition energy. Table B-4 contains the two air temperatures and the three corresponding relative humidities that were selected.

Table B-4. Environmental test conditions for Hartmann apparatus

| Test condition | Air temp. (°C) | Rel. Humidity (%) |
|---|---|---|
| 1 | 24 | 2 |
| 2 | 24 | 50 |
| 3 | 24 | 76 |
| 4 | 52 | 2 |
| 5 | 52 | 39 |
| 6 | 52 | 58 |

Temperature Control

Air temperature inside of the air reservoir and the Lucite test chamber was raised to either 24°C or 52°C using fiberglass wrapped, nichrome heating ribbon. Electrical power to the heating ribbon was controlled using a variable autotransformer. Thermocouples placed inside of the air space of the air reservoir and Lucite tube monitored their air temperatures. Heating ribbon was wrapped around the base of the dust dispersion cup to accelerate its heating rate. Adjustments were continuously made to the a[illegible] transformer until the desired steady state air temperature was a[illegible]ved. Figures 3, 4 and 5 show the heated components in detail.

Relative Humidity Control

The relative humidities reported in this program were attained by syringing calculated amounts of water into the heated air reservoir and dust dispersion cup. After the water vaporized, the reservoir was discharged into the Lucite tube. A sample of air from the tube was withdrawn, using a syringe, and analyzed for water content in the Photovolt Aquatest II, automatic Karl Fischer titrator. This procedure was repeated until the results were reproducible. Fifteen reproducible trials were performed at each temperature and relative humidity condition before the dust samples were introduced into the apparatus. Therefore, the environmental test air conditions presented in this report represent experimentally measured quantities. The 2 percent relative humidity values were obtained by purging dry, bottled compressed air through the Hartmann apparatus for two minutes prior to activating the "fire" switch.

Test Procedure For Obtaining Calibrated Environmental Air Conditions

The ex[illegible]iment was started by turning on the following equipment:

1. Air pressure (cylinder of dry air)
2. Power supply
3. Heating tapes
4. Potentiometer (thermocouple readout)
5. Photovolt - Aquatest II

Adjustments to the following items were then made:

1. Air pressure (69 k Pa)

2. Power supply (adjust energy setting between 50 and 8000 mj)

3. Heating tapes (24°C or 52°C)

4. Potentiometer (read millivolt output of iron constantan thermocouples)

5. Photovolt-Aquatest II (ready to receive sample)

The following sequence of operations was performed for each test cycle:

1. Distilled water was syringed into the brass reservoir and pressurized to a final pressure of 69 k Pa at the desired temperature.

2. Distilled water was syringed into the brass dust dispersion cup of the Lucite Hartmann tube.

3. The Lucite tube was secured and the high voltage leads were connected to the electrodes.

4. Fifteen minutes was allowed to elapse for vaporization of the water to occur.

5. The "fire" switch on the power supply was activated.

6. An air sample was removed from the Hartmann tube using a syringe. This sample was injected into the liquid solution of the automatic titrator. The parts per million of water in the air sample was read directly from the titrator.

Minimum Ignition Energy Tests Under Controlled Environmental Air Conditions

All tests conducted on the energetic materials followed the procedures described above. The only differences were the introduction of a weighed amount of explosive sample after the 15 minute vaporization period had elapsed and the elimination of the sixth item (air sample removal).

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